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REPEATABILITY AND REPRODUCIBILITY FOR COAL PROXIMATE ANALYSES

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REPEATABILITY AND REPRODUCIBILITY FOR COAL PROXIMATE ANALYSES

(COMPARING ASTM METHOD E-691 WITH ASTM PUBLISHED VALUES FOR 21 CANADIAN COMMERCIAL COALS OF VARIOUS RANKS)

by

M. Farrell *

ABSTRACT

Experimental data for moisture, ash, volatile, sulphur and calorific analyses submitted by laboratories participating in CANMET`s Service Program for the Evaluation of Characterization Samples were subjected to statistical treatment using ASTM method E-691 to yield precision values presented for Canadian coals of various ranks. The experimental data were collected in accordance with the provisions of ASTM statistical methods E-691 and E-180. The data from laboratories claiming to employ the most recent edition of ASTM standard methods were utilized in the calculations. The ASTM approved method of calculating the statistics of repeatability and reproducibility produced results which are shown along with the ASTM accepted values of repeatability and reproducibilty for each analysis.

The CANSPECS data were are presented in a manner that will establish procedures to compile and compare analytical method data repeatability and reproducibility. As the volume of CANSPECS data from other measures of coal quality become statistically significant further reports will be written on this topic.

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RÉPÉTABILITÉ ET REPRODUCTIBILITÉ DES ANALYSES QUANTITATIVES APPROXIMATIVES POUR LE CHARBON

(COMPARAISON ENTRE LA MÉTHODE ASTM E-691 ET LES VALEURS PUBLIÉES PAR ASTM POUR 21 CHARBONS CANADIENS DE QUALITÉ COMMERCIALE ET DE RANGS VARIÉS)

par

M. Farrell*

RÉSUMÉ

Les données expérimentales des analyses de la teneur en eau, en cendres, en matières volatiles et en soufre, et du pouvoir calorifique, présentées par les laboratoires participant à l'évaluation des échantillons de caractérisation dans le cadre du Programme d'Aide du CANMET, ont été soumises à une étude statistique à l'aide de la méthode ASTM E-691 dans le but d'obtenir des valeurs de précision pour des charbons canadiens de rangs variés. Ces données expérimentales ont été cueillies suivant les dispositions des méthodes statistiques E-691 et E-180 de l'ASTM. Les données provenant des laboratoires déclarant utiliser les plus récentes méthodes basées sur les normes de l'ASTM ont servi aux calculs. Les méthodes approuvées par l'ASTM pour le calcul statistique de la répétabilité et de la reproductibilité ont donné les résultats indiqués pour chaque analyse avec les valeurs de répétabilité et de reproductibilité acceptées par l'ASTM.

Les données CANSPEC ont été présentées dans le but de permettre l'élaboration de procédures pour réaliser la compilation et la comparaison des données obtenues à partir des méthodes d'analyse de la répétabilité et de la reproductibilité. À mesure que la quantité de données provenant d'autres mesures de la qualité du charbon, et recueillies par CANSPEC deviendra suffisamment importante pour une étude statistique, de nouveaux rapports seront préparés sur le sujet.

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INTRODUCTION

Analytical data for 21 air dried Canadian commercial coals of various ranks have been submitted by 50 laboratories from Canada and abroad. These data were subjected to statistical treatment by ASTM method E-691 to yield precision values.

CANMET`s Service Program for the Evaluation of Characterization Samples (CANSPECS) distributes carefully prepared, split and sealed Canadian commercial coal to 50 laboratories for analysis. The analyses are collated and a report is sent to each laboratory to enable them to assure the quality of their coal analysis. Under CANSPECS we ask for moisture, ash, volatile, sulphur and heating value analysis for each sample. The results are compiled and statistics calculated so each laboratory`s performance can be self monitored. These statistics also allow us the opportunity to present the repeatability and reproducibility values calculated with the ASTM(1) published values (obtained at each method`s acceptance or at its last revision). The data were collected in accordance with the provisions of E-691(2) and only the laboratories claiming to employ the most recent edition of ASTM standards were utilized in the calculations.

Rapid, inexpensive, quantitative determination of these fundamental parameters of coal quality is essential to efficient utilization in industrial processes. The use of standard methods and their attendant accuracy and precision are crucial to the correct evaluation of the quality of our coals and therefore the price and usefulness of our product.

The importance of this document is that it indicates coal analytical method's repeatability and reproducibility with a known confidence. This is important so we may determine the suitability of Canadian coals for various uses with a known confidence.

CANSPECS was recently expanded to include more analytical parameters of coal quality according to ASTM methods. The techniques described will be used in further determinations of these analysis confidence levels when the database for these analyses is significantly large.

ANALYTICAL PROCEDURES

The analytical procedures used to determine coal quality are standardized and published annually by the American Society for Testing and Materials. The ASTM book of standard testing methods encompasses the latest revisions produced by concensus by North American laboratories. Committee D5 on Coal and Coke meets semi-annually to discuss new methods and revisions to old to produce a document acceptable to coal producers, users and general interest groups. The data presented here were provided by participating laboratories consistently following the latest ASTM methods. These methods were modified slightly during the data gathering period from 1980 to 1986. Modifications are accepted on the basis that the analytical values derived by the new method concur with those from the old.

Participating laboratories do not all use the same methods and the statistical analysis of the results must reflect this. Thus a list was compiled of all the methods used from polling the participants during the last two years. Appendix 1 refers to laboratories by number (to retain their anonymity) with the method they used for each sample. These tables were used as the basis for selecting the data to generate precision values for each method.

The frequency and distribution of response to sample analysis request are also tabulated in Appendix 1 forming a cumulative total of the number of laboratories submitting analyses acceptable for the generation of precision statements for each method.

MOISTURE

Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled temperature, time, atmosphere, sample weight and equipment specifications. This method is outlined in greater detail as referenced in ASTM standard method D3173-73 (in force during this period). ASTM D3173 requires a 1 g sample of coal pulverized to pass a 250 um mesh to be accurately weighed in a standard size fused silica crucible placed in a minimum air volu furnace at 107C with a flow of dried air for 1 h.

ASH

Ash is determined by weighing the residue remaining after burning the coal under rigidly controlled conditions of sample weight, temperature, time, and atmosphere. This method is outlined in greater detail as referenced in ASTM standard method D3174-73 (in force during this period). ASTM D3174 requires a 1 g sample of coal pulverized to pass a 250 um mesh to be accurately weighed in a standard size fused silica crucible placed in a cool muffle furnace. Gradually heat the sample (too rapid expulsion of volatile matter can cause sample loss by mechanical loss) to 725C to a constant weight.

VOLATILE MATTER

Volatile matter is determined by establishing the weight loss resulting from heating a sample under rigidly controlled conditions. The measured weight loss, corrected for moisture as determined by Method D3173 establishes the volatile matter content. Two procedures are described to permit conformity with differences in sample behaviour. This method is outlined in greater detail as referenced in ASTM standard method D3175-77 (in force during this period). ASTM D3175 requires a 1 g sample of coal pulverized to pass a 250 um mesh to be accurately weighed in a covered platinum crucible placed into a furnace at 950C for precisely 7 min., then removed and allowed to cool for weighing. If the fuel does not yield a coherent cake as residue or sparks or an ash deposit on the crucible lid occurs the modified method must be used. The sparking fuel method entails a preliminary heating to 600C in 6 min. then heated in a furnace at 950C for 6 min.. The percentage loss in weight minus the per centage moisture as outlined in ASTM D3173 is the volatile matter content.

SULPHUR

Sulphur analysis is determined by burning a sample in a tube furnace at high temperature in a stream of oxygen to oxidize the sulphur in the coal under rigidly controlled conditions of sample weight, temperature, time, and atmosphere. The method most commonly used is outlined with greater detail as referenced in ASTM standard method D4239-83 option C (in force during this period). In this method one has three options A to C. These should give an incomparably close result in the context of this statistical process so the description below concentrates on the most widespread option C.

ASTM D4239 requires a 1 g sample of coal pulverized to pass a 250 um mesh to be accurately weighed. Analysis is performed by burning a sample in a tube furnace at a minimum temperature of 1350C in a stream of oxygen to oxidize the sulphur. Moisture and particulates are removed from the gas by traps filled with anhydrous magnesium perchlorate. The gas stream is passed through a cell in which sulphur dioxide is measured by an Infrared absorption detector. sulphur dioxide absorbs IR energy at a precise wavelength that is constantly monitored in the cell. This light is attenuated by the gas passing between the IR source and the detector within the cell. The attenuation of the transmitted wavelength specific to sulphur dioxide absorption from the time the gas of combustion first enters the cell until the gas is purged (the attenuation of the light drops to its former value) is summed. The magnitude of the sum is calibrated by combusting standard reference materials of known sulphur concentration spanning the range of interest. The calibration curve is checked for continuing accuracy during analysis of unknown concentration samples by assuring that samples of known concentration agree with their known values sufficiently.

CALORIFIC VALUE

Calorific value is determined by burning a weighed sample in an adiabatic oxygen bomb calorimeter under controlled conditions. The calorific value (or heating value) is calculated from temperature observations made before and after combustion, making proper allowance for thermometer and thermochemical corrections. This method is outlined in greater detail as referenced in ASTM standard method D2015-77 (in force during this period). ASTM D2015 requires a 1 g sample of coal pulverized to 250 um be weighed in an oxygen bomb ignition crucible. Add 1 mL of water to the bomb to absorb the nitrogen oxides for later titrimetric analysis using sodium carbonate (if significant nitrogen exists). A length of known calorific value wire is connected to the igniter electrodes with enough slack to permit the wire to contact with the sample. Pressurize the assembled bomb with 25 atm. of oxygen. Adjust the calorimeter water to 1.2C below room temperature. Transfer the bomb to the calorimeter, assure that it is gastight, and connect it to the firing circuit. Position the stirrers, thermometers and cover. Stir for 5 min. then correlate the jacket temperature to that of the calorimeter to within 0.01C and maintain for 3 min.. Record this initial temperature and ignite the charge. Maintain the jacket and calorimeter temperature difference as small as possible during the temperature rise and hold this difference to 0.01C when approaching the final equilibrium temperature. Take calorimeter temperature readings at 1 min. intervals until the same temperature is observed in three successive readings. Record this as the final temperature.

Remove the bomb and release the pressure slowly and evenly. If the bomb`s interior is sooty or some sample is unburned the analysis must be repeated. Wash the interior of the bomb with Methyl Red indicator in distilled water solution until the washings are acid free. Titrate this solution with sodium carbonate solution to determine the amount of nitrogen oxides produced.

Remove and measure or weigh the remaining ignitor wire to calculate the calorific value of the combusted wire.

Determine the sulphur content of the fuel using method D4283 in order to calculate the calorific value of the sulphur`s combustion.

The details of the method and calculations to correct for sulphur, nitrogen content in the fuel as well as the ignitor wire contribution, calibration, and standardization are described in the ASTM method D2015.

RESULTS

The CANSPECS samples were chosen to provide a wide spectrum of coal qualities and geographic locations. They were sampled and split in accordance with methods ASTM D346 and D2013. The analyses were reported on an as received basis according to D3180. These results were tabulated to display all raw data in an easily reviewed format. The coals were ranked by the suppliers of the material according to method D388. Each CANSPECS sample was analyzed by up to 50 laboratories in duplicate on two separate days. In accordance with the standard methods of statistical evaluation E691, E179, and E180 the results produced a grand mean, repeatability and reproducibility. The methods most often used by the participating laboratories were used for statistical comparison (because of the large database accumulated). The methods used by each laboratory are noted in Appendix 8 as tables 8-1 to 8-5 for each analysis. If the method was not indicated the laboratory's results were excluded. If a method was indicated we assumed it was used on the other samples we had submitted unless informed otherwise. The laboratories referred to by number to preserve their anonymity and to allow us to use the data in an unbiased manner.

When a laboratory`s analysis is found to have an average variation from one run to the next on the same day greater than the repeatability calculated we have 99% confidence in saying that this laboratory has too great a variation for the laboratory`s analysis to be valid. If a laboratory`s analysis is found to have a variation between its average greater than the reproducibility value away from the grand mean we have 99% confidence in saying that this laboratory has too great a variation for the method or analyzer to be operating correctly. Thus any laboratory whose repeatability and reproducibility exceeded the calculated excluded from the second calculation of grand mean, repeatability and reproducibility.

The grand mean for each CANSPECS sample is thus the analytical value around which most laboratory`s analytical values fell closest. The E691 repeatability is the value outside which each laboratory`s analysis result on repeating the same sample in the same laboratory with the same equipment should not exceed. The E691 reproducibility is the value outside which any laboratory`s analysis on analyzing the same sample in a different laboratory with different equipment should not exceed.

The accepted value for repeatability and reproducibility is the value that was agreed upon at the development or revision of each ASTM standard method and published in the manual of coal and coke testing methods. This value is generally accepted as an estimation. The data necessary for a close estimation of the repeatability and reproducibility must be at least as extensive as the CANSPECS data used to calculate the values presented here. (Note: The CANSPECS data comprise up to 50 laboratories analyzing the 21 separate samples 4 times for each of the 5 testing method results discussed.)

It is accepted that any measure of precision is directly related to the magnitude of the quantity measured. This is evidenced in the ASTM methods mentioned for the analyses studied here. For example if the moisture value is greater than 5% the repeatability increases from 0.2% to 0.3% and the reproducibility from 0.3% to 0.5%. Thus Fig. 1 to 10 display the grand mean values versus the E691 repeatability and reproducibility allowing us to easily see if any outlying values are present or if visible trends exist.

Tables 1 to 5 give the results on the 21 samples. A note at the bottom of each table explains the source of the repeatability and reproducibility accepted values shown.

Most of the coal produced and consumed in North America each year is on an as received basis as defined in ASTM D3180. It is important to the industry that the precision of the analytical methods be known on an as received basis so all the data and all the calculations and comparisons are on this basis. This entails a coupling of imprecisions as in the moisture analysis subtraction in the volatile method or the sulphur correction in the calorific method.

DISCUSSION

The precision values in Tables 1 to 5 were calculated after a single pass rejection of outliers according to the statistical criteria outlined in ASTM methods E691 and E180.

A small part of the precision value calculated by E691 and presented here for volatile matter has the imprecision of the moisture analysis inherent in its value. The moisture determined by each laboratory on each sample is subtracted from the measured amount of all volatile matter under the testing conditions.

The precision data for the sulphur analysis suffers from the fact that only about 20 laboratories generated acceptable analysis. At this number of data points the assumptions of E691 and E180 of a normal distribution for these data is strained. Method D3177 was used by over 10 laboratories for sulphur analysis during this period thus these results could not be used used to generate a precision statement based on ASTM D4239.

The calorific value is on an `as received` basis as defined by the ASTM calculated by D3180. This means that the precision values presented on this basis have inherent imprecisions due to other analytical methods. The air drying moisture loss can be considered negligable in the case of these samples. The calorific value has a correction for the heat of combustion of sulphur. Since the sulphur determination has an associated analytical error then the heat value repeatability and reproducibility have an error contribution from this source.

The within lab precision (repeatability) may be biased to smaller values as some laboratories may retest the coals until they meet an "expected" level of repeatability. The analysis repeatability measured may therefore be somewhat smaller than the actual case.

To observe more easily any trends in the repeatability and reproducibility values calculated by ASTM method E691 and the grand mean concentration these are plotted against one another in Figure 1 to 10.

The repeatability values presented are determined using the difference between the average of the two runs on the first day and the second day's.

The reproducibility values are calculated from the average difference between the grand mean of all the results and the average of one lab`s results.

An anomalous result is excluded from the final statistical output the first calculation will reject this lab on the basis of its repeatability or reproducibility being greater than the calculated threshold. This rejection of outlying laboratory results leaves all the other results to be included in the final determination of the repeatability or reproducibility.

This process produces the final statistics noted in Table 1 to 5. If one now looks at the tables one sees an occassional anomalously high repeatability or reproducibility. In order to determine if these anomalies are produced by a statistical method loophole we now inspect the data from these laboratories. It is then possible to ascertain the effect of deleteing this laboratory by reexecuting the final statistical output without this result. Thus a trial calculation of repeatability and reproducibility with candidate outliers deleted may indicate whether the deletions are valid. This process is shown in Appendix 1 through 8.

From Figure 1 all repeatability values for moisture are below 0.2% except that at 0.57%. From Fig. 2 all reproducibility values are below 1.5% except for this same sample with a reproducibility of 2.29%. From the raw data in Appendix 1 it is evident that the range of laboratories 16 and 3 and the difference of lab 23 results are markedly high. These samples were sent to the laboratories in sealed glass ampoules that may have been broken during delivery or by poor sample splitting. If we delete these data from the analysis, the repeatability and reproducibility become 0.38% and 1.77%. These values are calculated from a data set with 30 labs included and are more in line with the other values.

The repeatability value for ash in Fig. 3 shows a cluster of values with those of ash repeatability on an as received basis at or below 0.2% except for a low volatile bituminous sample with 0.3%. On closer inspection of these data in Appendix 2 one may see that laboratory 42 submitted anomalous data. This laboratory had the highest average of all largely because day one results average 15.00% and day two 13.20%. This sample was sent out in glass ampoules where the second day analysis must have been done on the contents of another glass ampoule. If one assumes that the first ampoule produced a high ash value (perhaps because of broken glass in the sample) we may delete this result. On deletion the repeatability is reduced from 0.30% to 0.16% (within range of the others) and the reproducibility is reduced from 0.73% to 0.47%.

The reproducibility values of ash in Fig. 4 shows all calculated reproducibilities below 1.0 except one. On closer inspection of the data producing the singularly high value of 1.52% we find one laboratory with an average of 24.17 whereas the rest of the lab's average is about half at 10.7. Assuming this lab had a calculation or mixing problem with this sample and delete their results from the database the reproducibility becomes 0.48. This value is well within the cluster of the other reproducibilities and the calculation results of this operation are set out in Appendix 3. The results show that one may express the reproducibility as a threshold value of 1.0%.

Figure 5 shows all repeatability values for volatile matter at 0.6% except for one at 0.78%. On inspection of the statistical analysis of the sample producing this value we find two laboratories which reported anomalous results. Lab 9 reported an average of 71.7% whereas the other labs averaged 37.2 and lab 35 first day results averaged 32.53% and the second day at 13.04%. If we delete these results and repeat the statistical calculations according to E691 the repeatability and reproducibility become 0.42% and 6.01% respectively (Appendix 4). The ASTM accepted way of expressing volatile repeatability is in descending values according to ascending rank. The values presently supported are listed in Table 3 along with the calculated values, which are all within the published repeatabilities.

The reproducibility values of volatile matter in Fig. 6 show all values below 2.5% when the volatile concentration is below 30%. Above a grand mean concentration of 30% to our highest value at 36.08% the reproducibilities show a seemingly exponential increase to reproducibilities more than the present maximum acceptable reproducibility of 2.0%. The highest reproducibility of 21.44% becomes 13.35% when the laboratory that reported 85.18%, versus an average of 31.6%, is deleted from the calculations (portrayed in Appendix 5). The third highest reproducibility of 10.60% becomes 6.01% as outlined in Appendix 4.

The second highest reproducibility is 12.51% on a Lignite A sample. Its raw data are displayed in Appendix 6 where the effect of deleting the lab reporting an average of 58.62% versus an average of 34.2% is deleted. The reproducibility decreases from 12.51% to 11.45%.

ASTM currently supports the concept that volatile statistical values are related to coal rank as noted in Table 3. If we artificially number each rank and attempt a linear regression with the repeatability and reproducibility a correlation may occur. The calculation assumptions and results are noted in Appendix 7 showing a coefficient of determination of 0.126 and 0.802 for repeatability and reproducibility respectively. The low value for correlation of the volatile repeatability shows the link to the coal rank may be tenuous. The relatively large coefficient for reproducibility shows that correlating the reproducibility of volatile matter with the rank of the coal is valid.

The repeatability value of sulphur in coal (Fig. 7) demonstrates all repeatability values are below 0.12%. The coefficient of determination for the linear regression analysis is 0.815 showing that a correlation exists between the concentration and the repeatability. This fact is acknowledged by ASTM method D4239 by an equation for calculating the sulphur repeatability.

The reproducibility value of sulphur given in Fig. 8 shows a strikingly visible linearity which is quantified with a coefficient of determination of 0.938. This finding confirms the present ASTM method of expressing the reproducibility as a linear equation of concentration.

The repeatability of heat value by D2015 versus grand mean values portrayed in Fig. 9 shows an even scatter with all values below 62 BTU/lb and most below 50 BTU/lb (as outlined in the method`s notes on repeatability). The linear regression analysis shows a very low correlation between heat value and repeatability as one would expect from the analytical confounding of the sulphur correction necessary for reporting the `as received` values requested and the weighing error from the moisture determination.

The reproducibility of heat value (Fig. 10) again shows a very wide scatter with all values below 300 BTU/lb and most below 200 BTU/lb. The linear regression coefficient of determination is 0.015 showing that the present ASTM policy of stating a threshold value for reproducibility to be valid.

The D2015 published values for heat value repeatability and reproducibility are presented on a dry basis so the values listed in Table 5 are calculated to the `as received` basis by multiplying by (1-(grand mean moisture)/100).

There are two relevant forms of expressing the repeatability and reproducibility for the ASTM methods studied here. They are a formula calculating the repeatability or reproducibility as a function of measured value or as a threshold value below which one expects the repeatability or reproducibility to fall.

If errors are due mainly to technical limitations of the method then one expects these errors to be proportional to the property measured. In this case a spectrum of samples with their calculated statistical values plotted against the grand mean will produce a straight line. This line is estimated by linear regression where the deviation of the data points about the best fitting line is quantified by the coefficient of determination. The closer these points are to the best fit line the closer to unity is the coefficient of determination.

The statistical value versus the analytical grand mean value is fitted by linear regression to determine the best line for the data. In this case the repeatability and reproducibility are fit with respect to the sample's grand mean value. If the coefficient of determination for the best line that may be fitted is close to zero then a correspondance between the statistical value and the grand mean is unlikely. This phenomenon indicates that the errors produced by the technical limitations of the method are overwhelmed by errors from other sources. These sources may be determined with a very large database with many measured parameters of coal quality. Unfortunately this size of database only produces a seemingly random dispersing of points on the figures.

It is now possible to discuss the repeatability and reproducibility as calculated by ASTM E691 effectively by regarding each analysis in turn for both the above statistical measures.

The coefficient of determination for all analyses except sulphur and perhaps moisture is very small. This indicates that linear dependence on the grand mean value is unlikely. In this case determining a threshold limiting value for repeatability and reproducibility is valid.

Table 6 shows a significantly high coefficient of determination for the repeatability and reproducibility at 0.815 and 0.938 respectively for sulphur. Thus the present D4239 method for expressing these values as linear functions of the sulphur concentration seems valid.

From the linear regression results displayed in Table 6 the coefficient of determination is 0.617 and 0.733 for repeatability and reproducibility for the moisture determination by ASTM D3173. This means we may express the repeatability and reproducibility as a function of moisture.

CONCLUSIONS

The moisture analysis as set out in ASTM D3173 describes the repeatability and reproducibility values as a function of concentration by stating that if the analytical value rises above 5% then the repeatability rises from 0.2% to 0.3% and the reproducibility from 0.3% to 0.5%. From these data the statistical measures may be more accurately stated as a linear function of concentration as follows;

Repeatability = (0.010) (analytical value) + 0.006

Reproducibility = (0.04) (analytical value) + 0.35

The ash analysis statistical limits as set out in D3174 allow almost all E691 calculated repeatability and reproducibility values to fall within the prescribed limits. The repeatability limits may be revised to a threshold value of 0.25% for all concentrations from 12 to 30% according to these data and their interpretation. The reproducibility value for ash should have a threshold value of 1.0% for all concentrations between 5 and 30%.

The volatile analysis as set out in D3175 prescribes repeatability and reproducibility according to coal rank. This seems more valid in the case of the reproducibility. The reproducibility values as found under the conditions of this sample analysis program appear much larger at low ranks than indicated in the present version of this method. A reproducibility of 15% on samples of more than 30% volatile is indicated. This conclusion departs significantly from the maximum acceptable 2.0% but there may be factors in this analysis that have been overlooked.

The sulphur analysis by D4239 presents a method of calculating the repeatability and reproducibility as a linear function of concentration which has been confirmed here. The repeatability equation adequately covered the E691 generated repeatabilities calculated here. The reproducibilities as calculated here are slightly larger than those set out in the present equation. From these data the statistical values may be stated as a linear function of concentration as follows;

Repeatability = (0.011) (analytical value) + 0.015

Reproducibility = (0.11) (analytical value) + 0.04

The heat value analysis by D2015 suggests a threshold repeatability of 50 BTU/lb which is confirmed by these data. D2015 also suggests a reproducibility of 100 BTU/lb which seem to be half the value necessary to account for the greatest part of the data. Note that these results are on the `as received` basis and not on the air dry basis that the present statistical values quoted above refer to so that the extra reproducibility and repeatability associated with the moisture determination should be included (here it was not).

The linear regression results for the 21 samples used here with an average of 30 laboratories responding with 4 results confirm the use of a concentration dependent equation for sulphur repeatability and reproducibility. These data also suggest the possibility of using this calculation format of expressing repeatability and reproducibility for moisture. The use of threshold values for the other analyses statistical values is confirmed.

CANMET`s Service Program for the Evaluation of Characterization Samples continues to operate with an expanded suite of analyses now including free swelling index, carbon, hydrogen and nitrogen. When the database from samples with adequate response to these analyses becomes available a more accurate determination of the repeatability and reproducibility of these standard ASTM methods will be possible. Conclusions were not more definitive due to the size of the database.

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The hundreds of technicians across North America who carefully analyzed each sample according to ASTM methods and presented the results in an unbiased manner were crucial to this report`s validity and the continuing success of CANSPECS.

Coal analytical laboratory supervisors who realized the usefulness of CANSPECS in maintaining their high quality of analysis and who responded promptly to analytical requests are appreciated.

Special thanks are given to Paul Bogardus of Tri-State Testing in Ashland, Kentucky as head of the ASTM statistical task group for coal and coke testing methods for reviewing the statistical accuracy of the statements.

REFERENCES

1. American Society for Testing of Materials
1985 Annual Book of ASTM Standards

2. Louis Janke; computer program & private communication.

Table 1 - Moisture analysis by ASTM D3173

Coal rank	Number of labs.	Moisture %	Repeatability		Reproducibilty	
			by E691	Accepted	by E691	Accepted
Lignite A	29	27.27	0.21	0.3	0.97	0.5
Lignite A	39	24.20	0.20	0.3	1.17	0.5
Subbit. C	38	15.97	0.17	0.3	0.91	0.5
Subbit. B	29	10.52	0.10	0.3	0.98	0.5
Subbit. B	33	28.81	0.57	0.3	2.29	0.5
H.V. C Bit.	25	7.74	0.11	0.3	0.81	0.5
H.V. C Bit.	38	2.88	0.10	0.2	0.63	0.3
H.V. C Bit.	37	3.14	0.08	0.2	0.33	0.3
H.V. C Bit.	41	6.36	0.10	0.3	0.63	0.5
H.V. B Bit.	43	8.60	0.13	0.3	0.75	0.5
H.V. B Bit.	37	0.68	0.06	0.2	0.21	0.3
H.V. A Bit.	30	0.44	0.07	0.2	0.34	0.3
H.V. A Bit.	40	1.34	0.08	0.2	0.50	0.3
H.V. A Bit.	35	2.93	0.13	0.2	0.45	0.3
M.V. Bit.	25	0.76	0.06	0.2	0.47	0.3
M.V. Bit.	31	0.58	0.04	0.2	0.27	0.3
M.V. Bit.	36	2.92	0.09	0.2	0.33	0.3
M.V. Bit.	42	0.87	0.05	0.2	0.27	0.3
M.V. Bit.	41	3.69	0.13	0.2	0.40	0.3
L.V. Bit.	25	0.88	0.07	0.2	0.53	0.3
L.V. Bit.	29	0.84	0.16	0.2	0.60	0.3

Note: If the moisture value rises above 5% the repeatability rises to 0.3% from 0.2% and the reproducibility to 0.5% from 0.3%.

Table 2 - Ash Analysis by ASTM D3174

Coal rank	Number of labs.	Ash %	Repeatability		Reproducibilty	
			by E691	Accepted	by E691	Accepted
Lignite A	27	10.30	0.11	0.3	0.67	0.5
Lignite A	38	10.79	0.16	0.3	1.52	0.5
SubBit. C	38	12.59	0.10	0.5	0.64	1.0
SubBit. B	28	16.08	0.09	0.5	0.80	1.0
SubBit. B	33	6.26	0.14	0.3	0.66	0.5
H.V. C Bit.	24	10.26	0.06	0.3	0.36	0.5
H.V. C Bit.	37	11.24	0.11	0.3	0.30	0.5
H.V. C Bit.	37	17.91	0.12	0.5	0.38	1.0
H.V. C Bit.	40	11.47	0.09	0.3	0.22	0.5
H.V. B Bit.	42	9.09	0.07	0.3	0.37	0.5
H.V. B Bit.	36	9.96	0.08	0.3	0.23	0.5
H.V. A Bit.	29	19.47	0.20	0.5	0.54	1.0
H.V. A Bit.	40	8.65	0.09	0.3	0.28	0.5
H.V. A Bit.	34	23.43	0.19	0.3	0.49	0.5
M.V. Bit.	24	6.75	0.14	0.3	0.29	0.5
M.V. Bit.	30	12.99	0.09	0.5	0.26	1.0
M.V. Bit.	35	19.47	0.10	0.5	0.29	1.0
M.V. Bit.	41	15.87	0.14	0.5	0.36	1.0
M.V. Bit.	40	26.79	0.17	0.5	0.58	1.0
L.V. Bit.	24	6.31	0.09	0.3	0.49	0.5
L.V. Bit.	30	13.03	0.30	0.5	0.73	1.0

Note: If the ash value rises above 12% the repeatability rises to 0.3% from 0.2% and the reproducibility to 1.0% from 0.5% (assuming that both carbonates and/or pyrites are present)

Table 3 - Volatile Analysis by ASTM D3175

Coal rank	Number of labs.	Volatile %	Repeatability		Reproducibilty	
			by E691	Accepted	by E691	Accepted
Lignite A	27	34.83	0.33	1.0	21.44	2.0
Lignite A	35	34.23	0.44	1.0	12.51	2.0
SubBit. C	38	35.31	0.78	1.0	10.60	2.0
SubBit. B	28	32.42	0.38	0.7	6.11	1.4
SubBit. B	30	33.44	0.55	0.7	7.38	1.4
H.V. C Bit.	23	36.08	0.35	0.5	5.06	1.0
H.V. C Bit.	35	34.93	0.38	0.5	3.58	1.0
H.V. C Bit.	35	20.40	0.22	0.5	1.30	1.0
H.V. C Bit.	38	32.88	0.42	0.5	2.33	1.0
H.V. B Bit.	40	33.44	0.34	0.5	2.98	1.0
H.V. B Bit.	35	24.56	0.19	0.5	1.16	1.0
H.V. A Bit.	29	34.70	0.29	0.5	2.10	1.0
H.V. A Bit.	38	34.51	0.43	0.5	1.91	1.0
H.V. A Bit.	33	30.93	0.31	0.5	1.46	1.0
M.V. Bit.	24	21.08	0.35	0.5	2.36	1.0
M.V. Bit.	30	20.26	0.23	0.5	1.21	1.0
M.V. Bit.	35	20.27	0.32	0.5	1.48	1.0
M.V. Bit.	39	21.08	0.24	0.5	1.23	1.0
M.V. Bit.	38	29.63	0.34	0.5	1.77	1.0
L.V. Bit.	24	13.77	0.19	0.5	1.38	1.0
L.V. Bit.	29	27.57	0.48	0.5	1.56	1.0

Note: Repeatability and reproducibility are related to coal rank: bituminous 0.5,1.0; subbituminous 0.7,1.4; and lignite 1.0,2.0 respectively.

Table 4 - sulphur analysis by ASTM D4239

Coal rank	Number of labs.	sulphur %	Repeatability		Reproducibilty	
			by E691	Accepted	by E691	Accepted
Lignite A	17	0.37	0.01	0.04	0.11	0.07
Lignite A	26	0.40	0.03	0.05	0.11	0.07
SubBit. C	24	1.05	0.05	0.07	0.16	0.11
SubBit. B	19	0.38	0.02	0.05	0.12	0.07
SubBit. B	21	0.47	0.03	0.05	0.11	0.08
H.V. C Bit.	16	0.82	0.02	0.06	0.16	0.10
H.V. C Bit.	23	6.23	0.12	0.28	0.78	0.42
H.V. C Bit.	24	0.28	0.02	0.04	0.10	0.07
H.V. C Bit.	27	0.24	0.01	0.04	0.09	0.06
H.V. B Bit.	27	0.28	0.01	0.04	0.09	0.07
H.V. B Bit.	22	0.40	0.02	0.05	0.09	0.07
H.V. A Bit.	18	9.36	0.11	0.40	1.16	0.61
H.V. A Bit.	27	2.81	0.04	0.14	0.27	0.22
H.V. A Bit.	20	3.96	0.04	0.19	0.35	0.29
M.V. Bit.	13	0.40	0.01	0.05	0.09	0.07
M.V. Bit.	18	0.21	0.02	0.04	0.06	0.06
M.V. Bit.	26	0.26	0.01	0.04	0.07	0.07
M.V. Bit.	27	0.29	0.02	0.04	0.08	0.07
M.V. Bit.	27	4.43	0.05	0.21	0.32	0.32
L.V. Bit.	17	0.71	0.02	0.06	0.18	0.09
L.V. Bit.	20	2.32	0.06	0.12	0.23	0.19

Note: Repeatability is calculated from the grand mean as 0.03+0.04(g.m.)
Reproducibility is calculated from the grand mean as 0.05+0.06(g.m.)

Table 5 - Heat Value Analysis by ASTM D2015

Coal rank	Number of labs.	Heat Value BTU/lb	Repeatability		Reproducibilty	
			by E691	Accepted	by E691	Accepted
Lignite A	27	7518	31	36	152	73
Lignite A	37	7657	23	42	210	84
SubBit. C	36	8500	32	38	152	76
SubBit. B	28	9090	29	45	108	89
SubBit. B	32	8295	40	36	253	71
H.V. C Bit.	24	10917	33	46	143	92
H.V. C Bit.	37	11761	34	49	158	97
H.V. C Bit.	35	11490	24	48	102	97
H.V. C Bit.	37	11064	48	47	191	94
H.V. B Bit.	42	11060	39	46	187	91
H.V. B Bit.	36	13878	33	50	112	99
H.V. A Bit.	28	12124	29	50	273	100
H.V. A Bit.	41	13291	37	49	161	99
H.V. A Bit.	34	9990	29	49	127	99
M.V. Bit.	23	14516	52	50	167	99
M.V. Bit.	30	13293	38	50	137	100
M.V. Bit.	35	11116	31	50	132	100
M.V. Bit.	41	12333	29	50	126	100
M.V. Bit.	40	9429	32	50	157	100
L.V. Bit.	24	14281	21	50	190	100
L.V. Bit.	31	13357	61	50	168	100

Note: The accepted repeatability and reproducibility are only stated on the dry basis as 50 and 100 BTU/lb respectively. The as received values are calculated from these on multiplying by (1-(Grand Mean Moisture)/100).

The statistical results may also be evaluated by performing a linear regression analysis to ascertain if any linear correspondance exists between the grand mean concentration of these samples and their calculated repeatability and reproducibility. The results of the standard linear regression routine are displayed in Table 6.

Table 6 - Linear regression of concentration versus repeat. and reprod.

Analysis	Statistic = (M +/- m) * (Mean Value) + (B +/- b)									
	Repeatability					Reproducibility				
	M	m	B	b	C of Det	M	m	B	b	C of Det
Moisture	.010	.004	.006	.004	0.617	0.04	0.01	0.3	0.1	0.733
Ash	.004	.004	.08	.06	0.140	.00	.03	0.5	0.4	0.006
Volatile	.012	.008	.02	0.2	0.346	0.4	0.3	-6.	9.	0.240
sulphur	.011	.003	.015	.008	0.815	0.11	0.01	0.04	0.04	0.938
BtU/lb	.001	.002	20.	20.	0.101	-.002	.009	190.	110.	0.015

Note: C of Det = Coefficient of Determination (unity at perfect correlation).

FIGURE 1

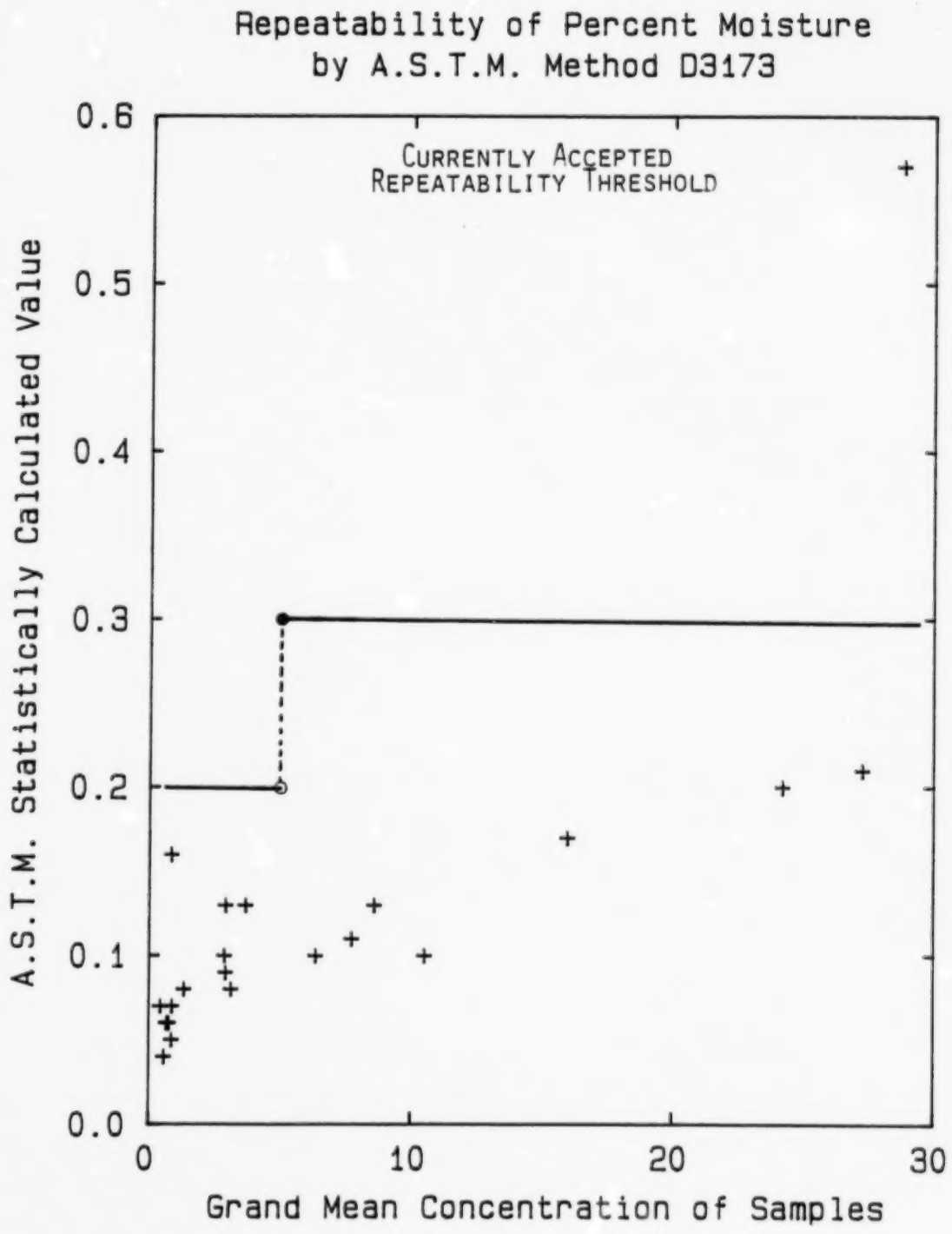

Repeatability of Percent Moisture
by A.S.T.M. Method D3173
CURRENTLY ACCEPTED
REPEATABILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
0.6
0.5
0.4
0.3
0.2
0.1
0.0
0
10
20
30
Grand Mean Concentration of Samples

FIGURE 2

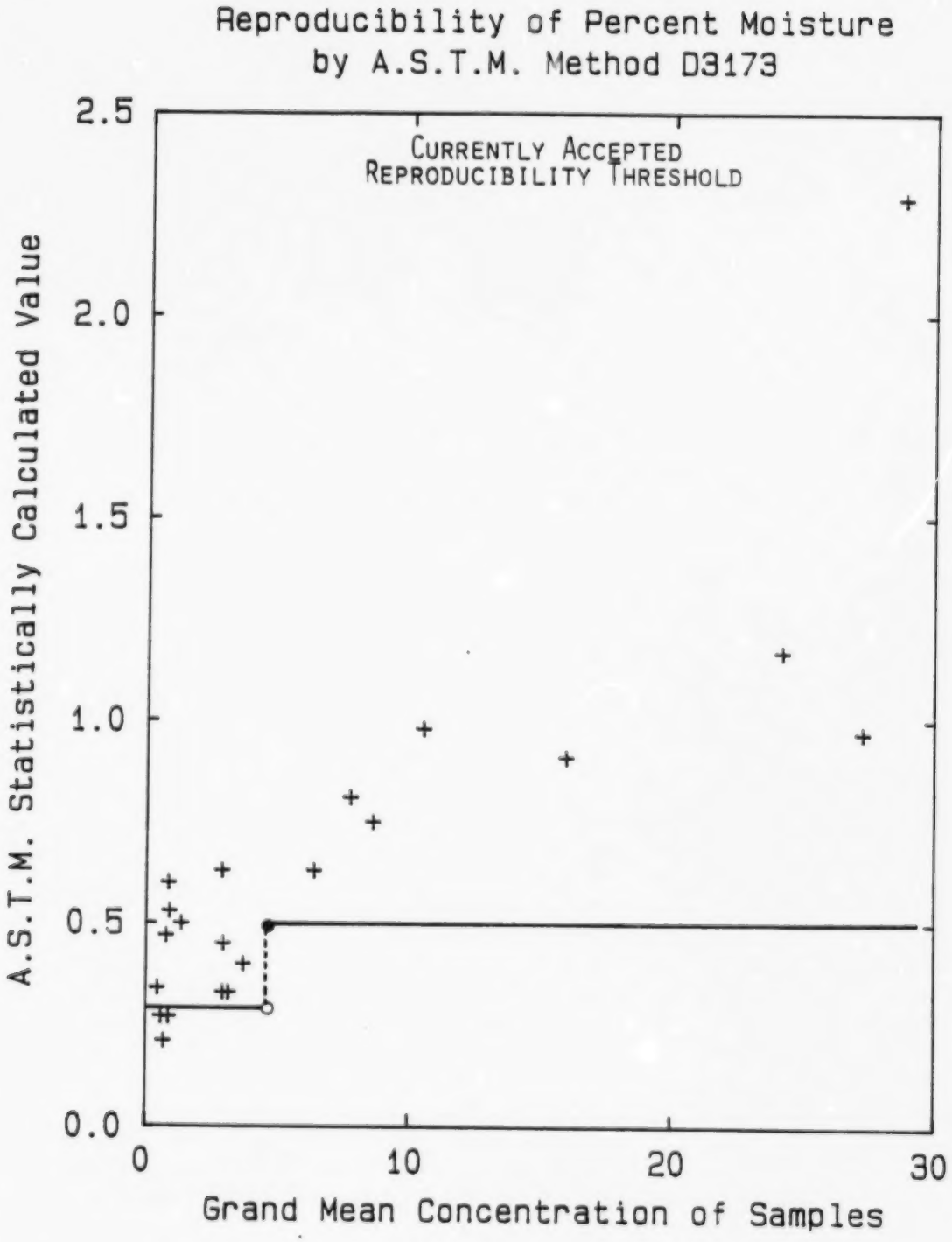

Reproducibility of Percent Moisture
by A.S.T.M. Method D3173
CURRENTLY ACCEPTED
REPRODUCIBILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
2.5
2.0
1.5
1.0
0.5
0.0
0
10
20
30
Grand Mean Concentration of Samples

FIGURE 3

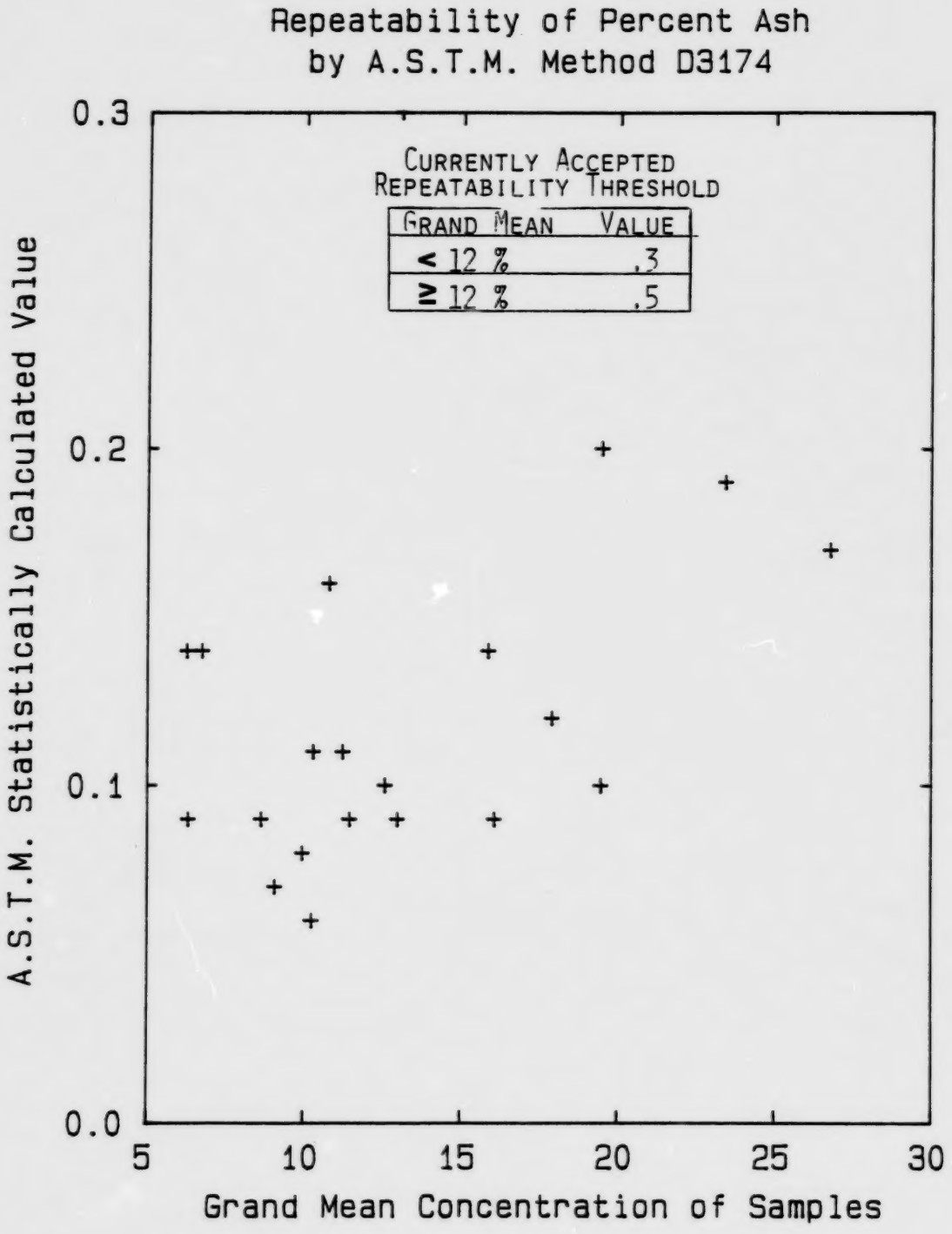

Repeatability of Percent Ash
by A.S.T.M. Method D3174
CURRENTLY ACCEPTED
REPEATABILITY THRESHOLD
GRAND MEAN VALUE
< 12 % .3
≥ 12 % .5
A.S.T.M. Statistically Calculated Value
0.3
0.2
0.1
0.0
5
10
15
20
25
30
Grand Mean Concentration of Samples

FIGURE 4

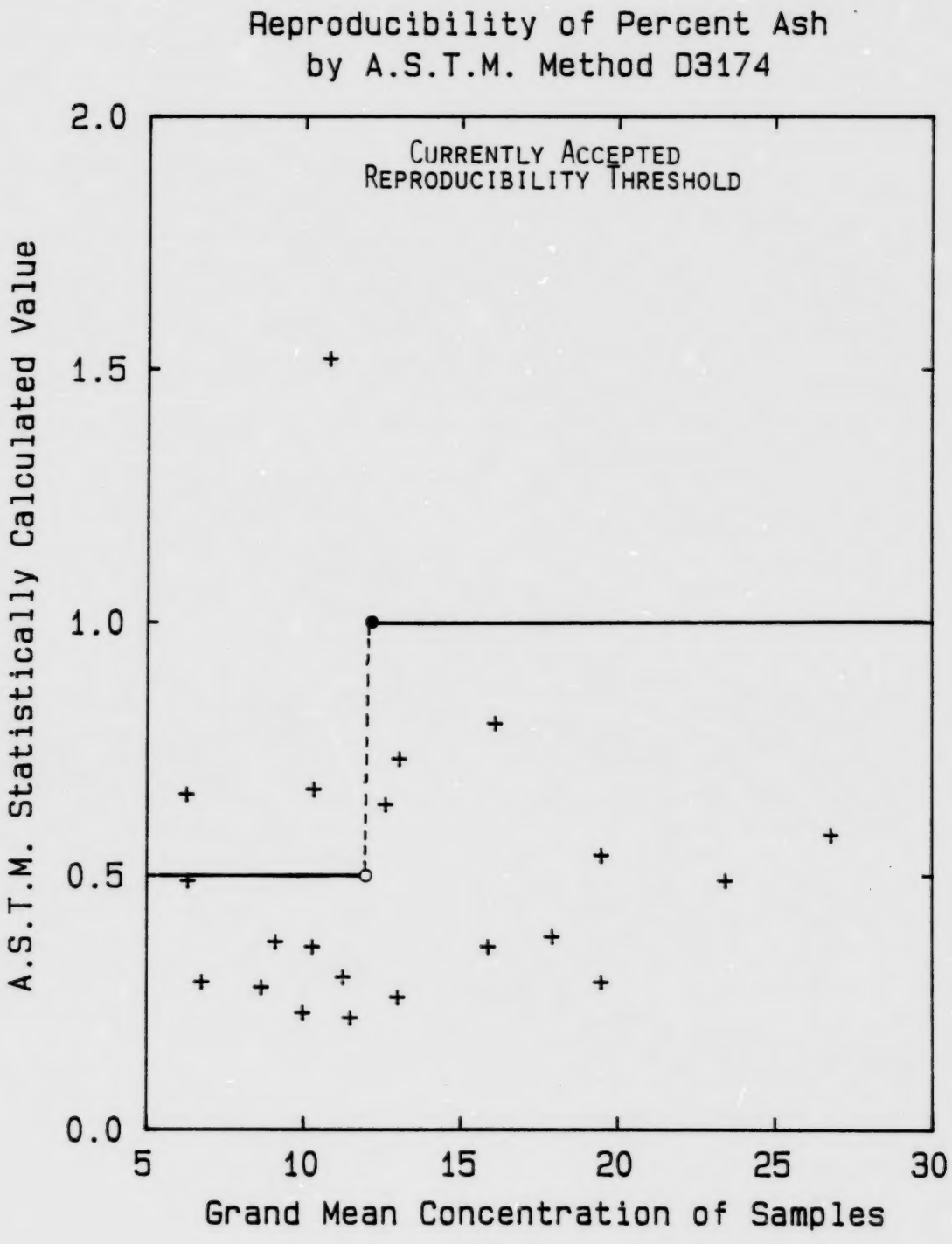

Reproducibility of Percent Ash
by A.S.T.M. Method D3174
CURRENTLY ACCEPTED
REPRODUCIBILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
2.0
1.5
1.0
0.5
0.0
5
10
15
20
25
30
Grand Mean Concentration of Samples

FIGURE 5

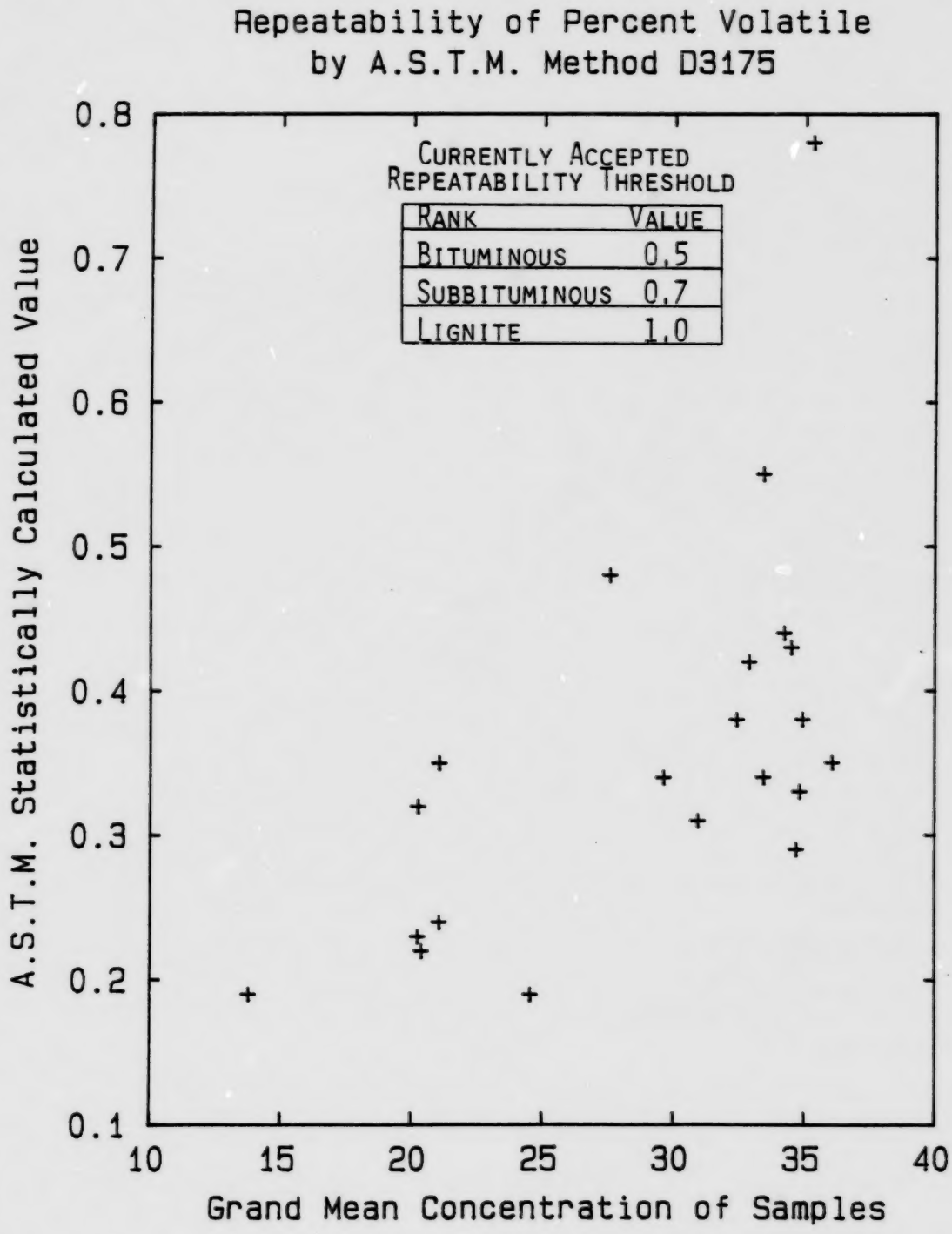

Repeatability of Percent Volatile
by A.S.T.M. Method D3175
CURRENTLY ACCEPTED
REPEATABILITY THRESHOLD
RANK VALUE
BITUMINOUS 0.5
SUBBITUMINOUS 0.7
LIGNITE 1.0
A.S.T.M. Statistically Calculated Value
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
10
15
20
25
30
35
40
Grand Mean Concentration of Samples

FIGURE 6

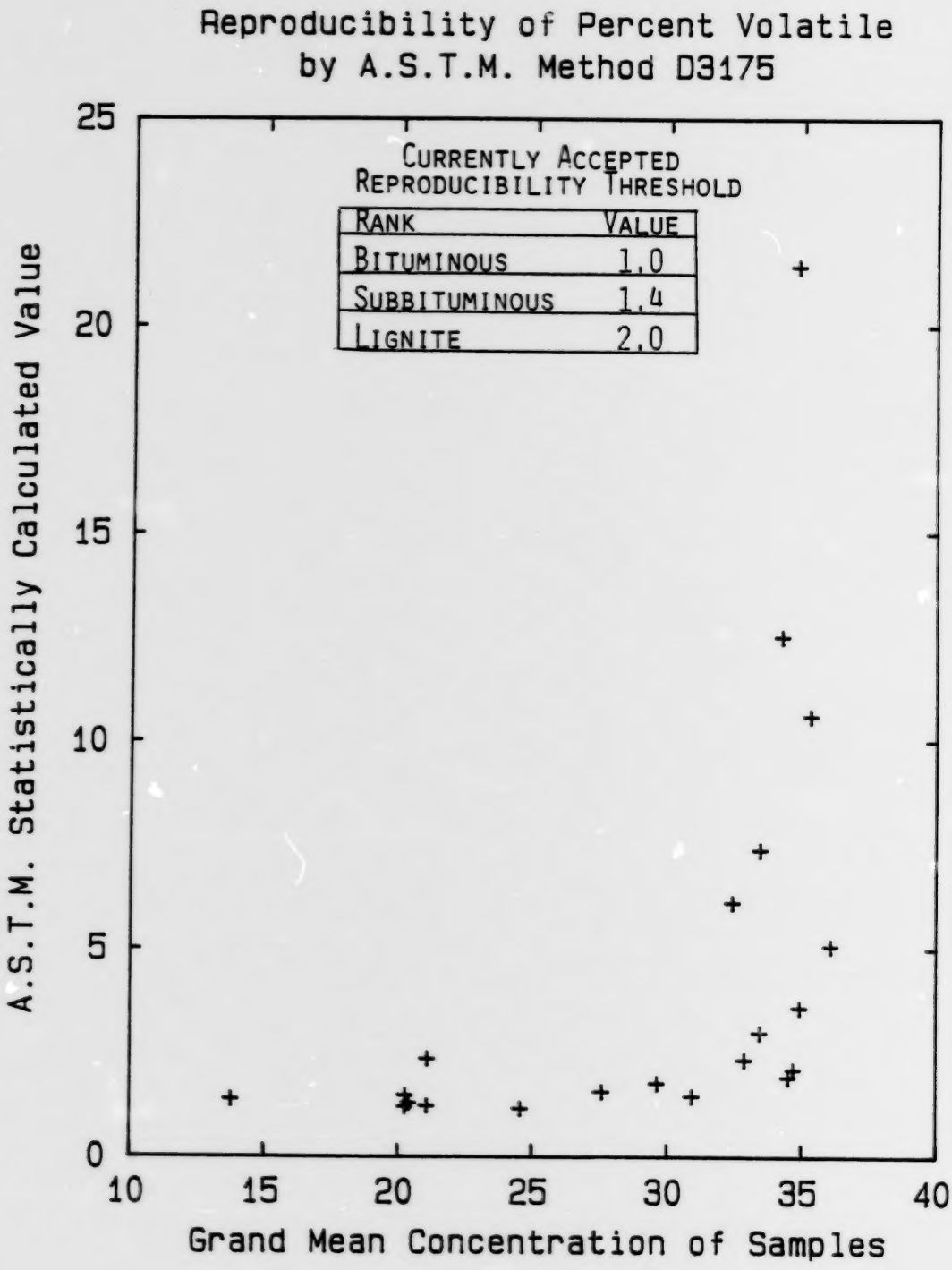

Reproducibility of Percent Volatile
by A.S.T.M. Method D3175
CURRENTLY ACCEPTED
REPRODUCIBILITY THRESHOLD
RANK VALUE
BITUMINOUS 1.0
SUBBITUMINOUS 1.4
LIGNITE 2.0
A.S.T.M. Statistically Calculated Value
Grand Mean Concentration of Samples
0
5
10
15
20
25
10
15
20
25
30
35
40

FIGURE 7

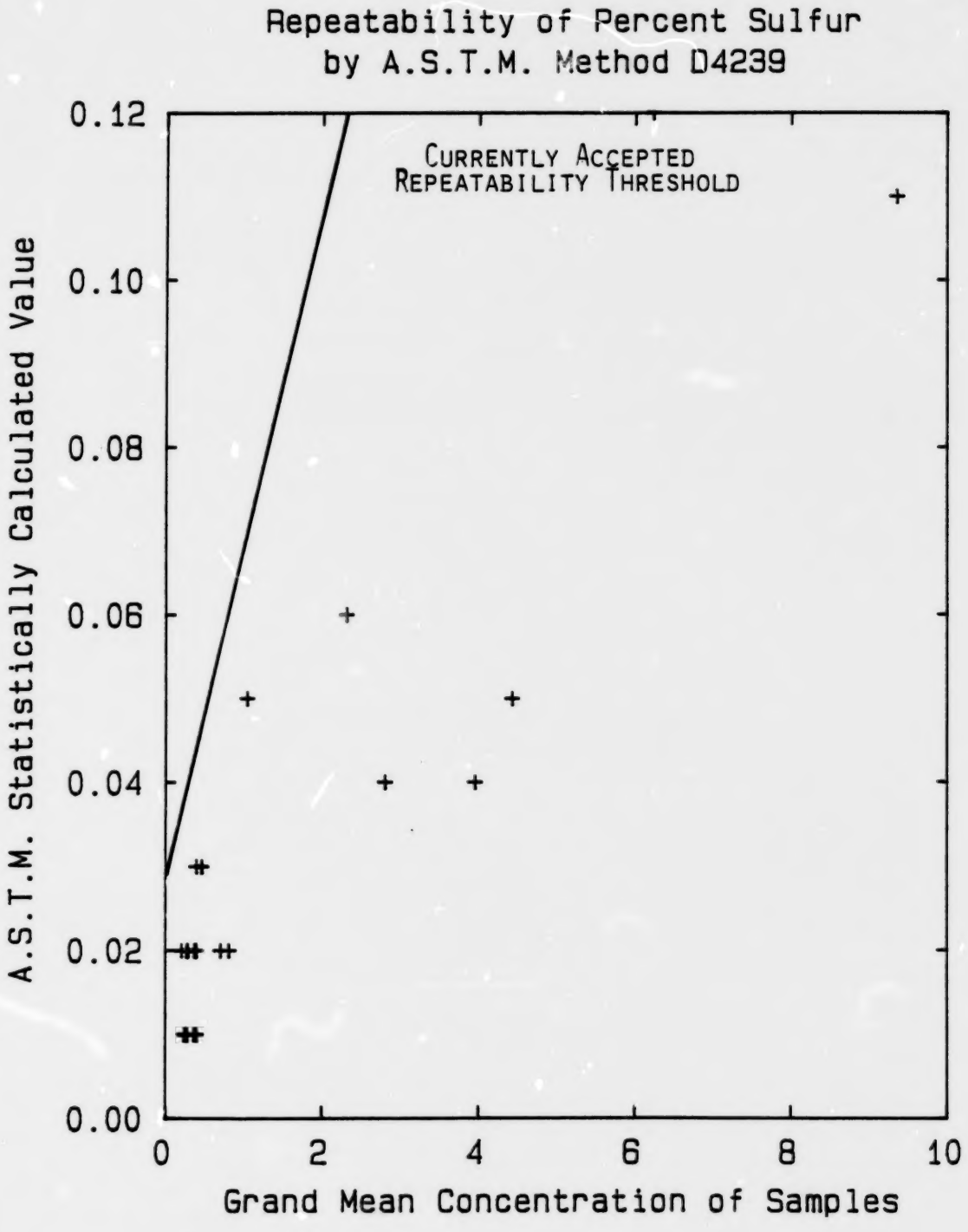

Repeatability of Percent Sulfur
by A.S.T.M. Method D4239
CURRENTLY ACCEPTED
REPEATABILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0
2
4
6
8
10
Grand Mean Concentration of Samples

FIGURE 8

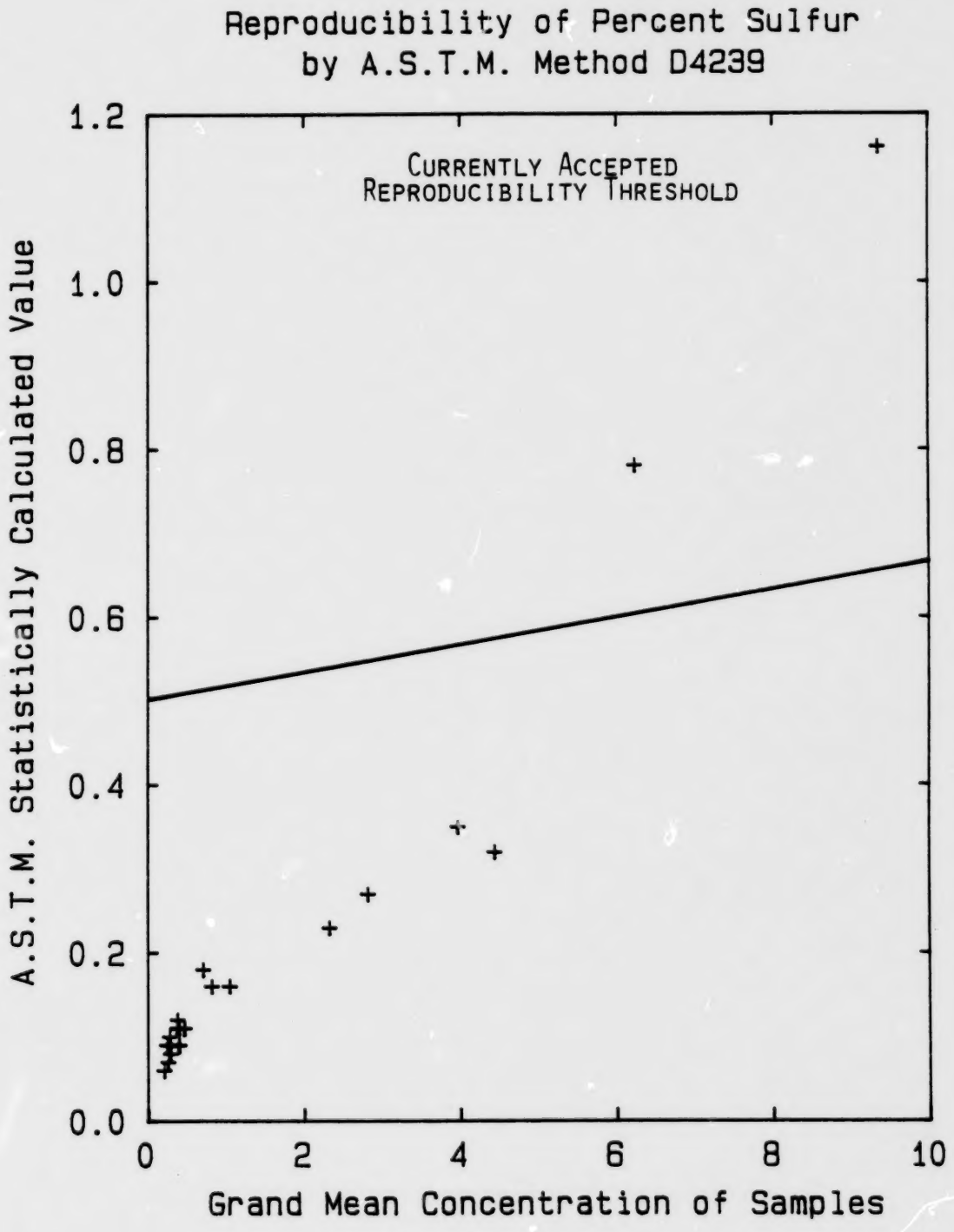

Reproducibility of Percent Sulfur
by A.S.T.M. Method D4239
CURRENTLY ACCEPTED
REPRODUCIBILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
1.2
1.0
0.8
0.6
0.4
0.2
0.0
0
2
4
6
8
10
Grand Mean Concentration of Samples

FIGURE 9

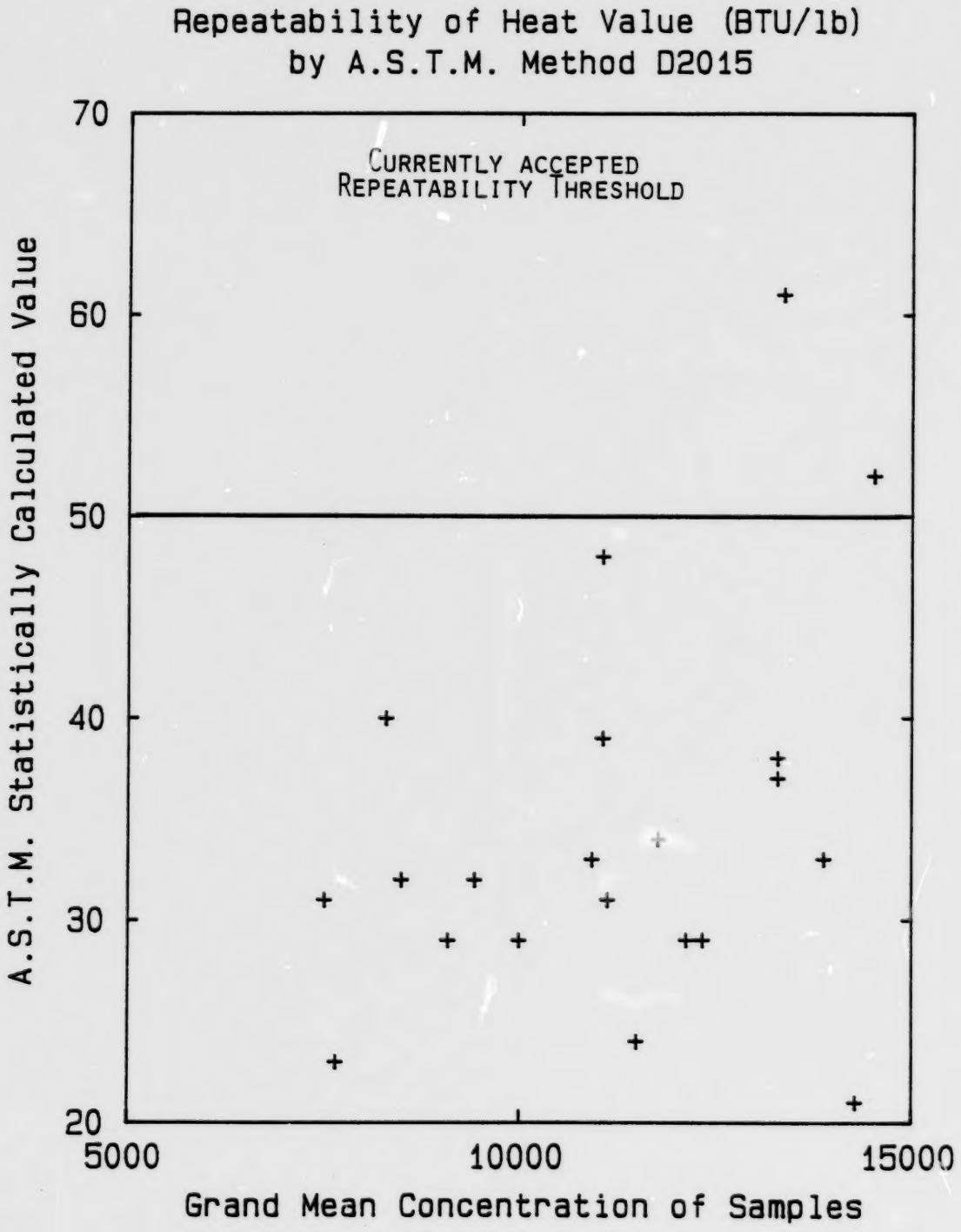
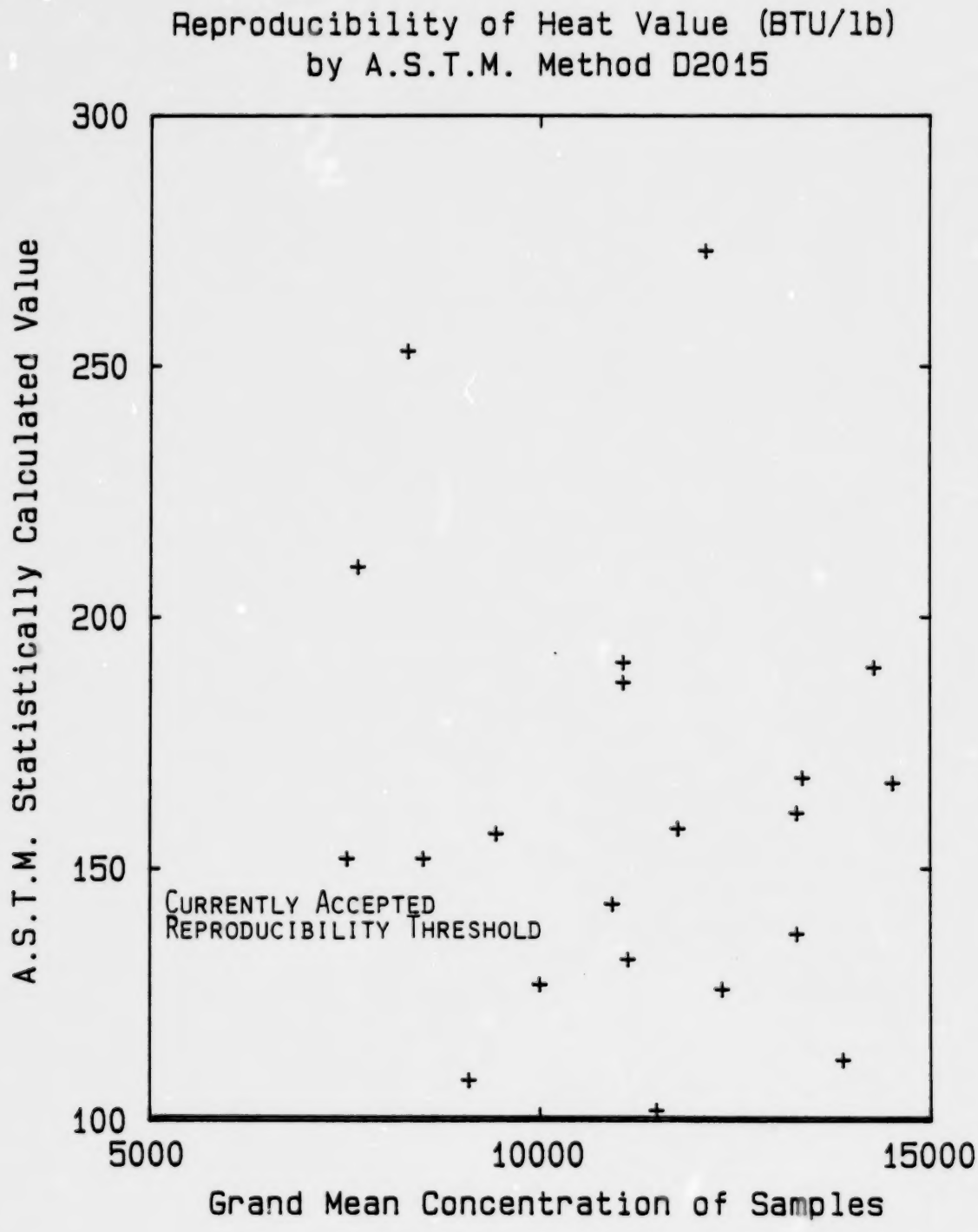

Repeatability of Heat Value (BTU/lb)
by A.S.T.M. Method D2015
CURRENTLY ACCEPTED
REPEATABILITY THRESHOLD
A.S.T.M. Statistically Calculated Value
70
60
50
40
30
20
5000
10000
15000
Grand Mean Concentration of Samples

FIGURE 10


Reproducibility of Heat Value (BTU/lb)
by A.S.T.M. Method D2015
A.S.T.M. Statistically Calculated Value
300
250
200
150
100
5000
10000
15000
Grand Mean Concentration of Samples
CURRENTLY ACCEPTED
REPRODUCIBILITY THRESHOLD

APPENDIX 1

Repeatability and reproducibility of per cent moisture for sample 23

=(0.57,2.29)

Delete results from laboratories 3, 16, 23

=(0.38,1.77)

CANSPECS
STATISTICAL ANALYSIS OF MOISTURE

33 of 50 reporting sample : 23

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
12	30.39	30.31	30.55	30.46	30.43	2.19	0.15	
15	30.39	30.40	30.41	30.37	30.39	2.15	0.01	
8	29.75	29.80	29.80	29.70	29.76	1.52	0.02	
45	29.98	29.80	29.79	29.40	29.74	1.50	0.29	
40	29.82	29.75	29.60	29.43	29.65	1.41	0.27	
34	29.65	29.65	29.63	29.63	29.64	1.40	0.02	
4	29.57	29.59	29.55	29.59	29.58	1.33	0.01	
18	29.22	29.22	29.76	29.69	29.47	1.23	0.51	
7	29.59	29.32	29.47	29.21	29.40	1.16	0.12	
37	29.32	29.25	29.33	29.55	29.36	1.12	0.15	
21	29.48	29.37	29.26	29.15	29.32	1.07	0.22	
48	29.41	29.48	29.06	29.18	29.28	1.04	0.32	
31	29.09	29.27	29.11	29.23	29.18	0.93	0.01	
35	29.19	29.12	29.17	29.17	29.16	0.92	0.01	
24	29.28	29.18	29.01	29.08	29.14	0.90	0.19	
33	29.25	29.25	29.00	29.00	29.13	0.88	0.25	
43	29.04	29.01	28.99	28.93	28.99	0.75	0.07	
30	28.84	28.90	29.11	28.68	28.88	0.64	0.02	
22	28.83	28.76	29.09	28.75	28.86	0.62	0.12	
39	28.72	28.76	28.77	28.78	28.76	0.52	0.04	
25	29.03	29.32	28.24	28.24	28.71	0.47	0.94	
44	28.84	28.63	28.70	28.65	28.71	0.46	0.06	
29	28.66	28.65	28.63	28.53	28.62	0.38	0.08	
38	28.65	28.83	27.98	27.98	28.36	0.12	0.76	
27	28.26	28.29	28.33	28.43	28.33	0.09	0.10	
			[GRAND MEAN]=		28.24	A= 4.63 C=	1.36	
20	28.01	28.20	28.15	28.15	28.13	0.11	0.04	
42	27.00	27.60	26.90	26.80	27.08	1.17	0.45	
13	26.73	26.91	25.98	26.53	26.54	1.70	0.56	
32	26.34	26.44	26.40	26.31	26.37	1.87	0.04	
47	25.72	25.82	24.97	24.88	25.35	2.89	0.85	
16	23.21	23.22	26.20	25.70	24.58	3.66	2.74	C *
3	26.87	25.48	24.07	21.76	24.55	3.70	3.26	C *
23	18.50	18.52	18.59	18.55	18.54	9.70	0.06	A *

CANSPECS
STATISTICAL ANALYSIS OF MOISTURE

33 of 50 reporting sample : 23

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
12	30.39	30.31	30.55	30.46	30.43	1.62	0.15	B
15	30.39	30.40	30.41	30.37	30.39	1.58	0.01	B
8	29.75	29.80	29.80	29.70	29.76	0.95	0.02	B
45	29.98	29.80	29.79	29.40	29.74	0.93	0.29	B
40	29.82	29.75	29.60	29.43	29.65	0.84	0.27	B
34	29.65	29.65	29.63	29.63	29.64	0.83	0.02	B
4	29.57	29.59	29.55	29.59	29.58	0.77	0.01	B
18	29.22	29.22	29.76	29.69	29.47	0.66	0.51	B D
7	29.59	29.32	29.47	29.21	29.40	0.59	0.12	B
37	29.32	29.25	29.33	29.55	29.36	0.55	0.15	B
21	29.48	29.37	29.26	29.15	29.32	0.51	0.22	B
48	29.41	29.48	29.06	29.18	29.28	0.47	0.32	D
31	29.09	29.27	29.11	29.23	29.18	0.37	0.01	
35	29.19	29.12	29.17	29.17	29.16	0.35	0.01	
24	29.28	29.18	29.01	29.08	29.14	0.33	0.19	
33	29.25	29.25	29.00	29.00	29.13	0.32	0.25	
43	29.04	29.01	28.99	28.93	28.99	0.18	0.07	
30	28.84	28.90	29.11	28.68	28.88	0.07	0.02	
22	28.83	28.76	29.09	28.75	28.86	0.05	0.12	
			[GRAND MEAN]=		28.81	A= 2.29	C= 0.57	
39	28.72	28.76	28.77	28.78	28.76	0.05	0.04	
25	29.03	29.32	28.24	28.24	28.71	0.10	0.94	CD
44	28.84	28.63	28.70	28.65	28.71	0.10	0.06	
29	28.66	28.65	28.63	28.53	28.62	0.19	0.08	
38	28.65	28.83	27.98	27.98	28.36	0.45	0.76	CD
27	28.26	28.29	28.33	28.43	28.33	0.48	0.10	
20	28.01	28.20	28.15	28.15	28.13	0.68	0.04	B
42	27.00	27.60	26.90	26.80	27.08	1.73	0.45	B D
13	26.73	26.91	25.98	26.53	26.54	2.27	0.56	B D
32	26.34	26.44	26.40	26.31	26.37	2.44	0.04	AB
47	25.72	25.82	24.97	24.88	25.35	3.46	0.85	ABCD
16	23.21	23.22	26.20	25.70	24.58	4.23	2.74	ABCD*
3	26.87	25.48	24.07	21.76	24.55	4.26	3.26	ABCD*
23	18.50	18.52	18.59	18.55	18.54	10.27	0.06	AB *

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (0.5)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.3)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF MOISTURE

30 of 50 reporting sample : 23

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
12	30.39	30.31	30.55	30.46	30.43	1.62	0.15	
15	30.39	30.40	30.41	30.37	30.39	1.58	0.01	
8	29.75	29.80	29.80	29.70	29.76	0.95	0.02	
45	29.98	29.80	29.79	29.40	29.74	0.93	0.29	
40	29.82	29.75	29.60	29.43	29.65	0.84	0.27	
34	29.65	29.65	29.63	29.63	29.64	0.83	0.02	
4	29.57	29.59	29.55	29.59	29.58	0.77	0.01	
18	29.22	29.22	29.76	29.69	29.47	0.66	0.51	
7	29.59	29.32	29.47	29.21	29.40	0.59	0.12	
37	29.32	29.25	29.33	29.55	29.36	0.55	0.15	
21	29.48	29.37	29.26	29.15	29.32	0.51	0.22	
48	29.41	29.48	29.06	29.18	29.28	0.47	0.32	
31	29.09	29.27	29.11	29.23	29.18	0.37	0.01	
35	29.19	29.12	29.17	29.17	29.16	0.35	0.01	
24	29.28	29.18	29.01	29.08	29.14	0.33	0.19	
33	29.25	29.25	29.00	29.00	29.13	0.32	0.25	
43	29.04	29.01	28.99	28.93	28.99	0.18	0.07	
30	28.84	28.90	29.11	28.68	28.88	0.07	0.02	
22	28.83	28.76	29.09	28.75	28.86	0.05	0.12	
			[GRAND MEAN]=		28.81	A= 2.29 C=	0.57	
39	28.72	28.76	28.77	28.78	28.76	0.05	0.04	
25	29.03	29.32	28.24	28.24	28.71	0.10	0.94	C *
44	28.84	28.63	28.70	28.65	28.71	0.10	0.06	
29	28.66	28.65	28.63	28.53	28.62	0.19	0.08	
38	28.65	28.83	27.98	27.98	28.36	0.45	0.76	C *
27	28.26	28.29	28.33	28.43	28.33	0.48	0.10	
20	28.01	28.20	28.15	28.15	28.13	0.68	0.04	
42	27.00	27.60	26.90	26.80	27.08	1.73	0.45	
13	26.73	26.91	25.98	26.53	26.54	2.27	0.56	
32	26.34	26.44	26.40	26.31	26.37	2.44	0.04	A *
47	25.72	25.82	24.97	24.88	25.35	3.46	0.85	A C *

CANSPECS
STATISTICAL ANALYSIS OFMOISTURE

30 of 50 reporting sample : 23

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
12	30.39	30.31	30.55	30.46	30.43	1.37	0.15	B
15	30.39	30.40	30.41	30.37	30.39	1.33	0.01	B
8	29.75	29.80	29.80	29.70	29.76	0.70	0.02	B
45	29.98	29.80	29.79	29.40	29.74	0.68	0.29	B
40	29.82	29.75	29.60	29.43	29.65	0.59	0.27	B
34	29.65	29.65	29.63	29.63	29.64	0.58	0.02	B
4	29.57	29.59	29.55	29.59	29.58	0.52	0.01	B
18	29.22	29.22	29.76	29.69	29.47	0.41	0.51	CD
7	29.59	29.32	29.47	29.21	29.40	0.34	0.12	
37	29.32	29.25	29.33	29.55	29.36	0.30	0.15	
21	29.48	29.37	29.26	29.15	29.32	0.26	0.22	
48	29.41	29.48	29.06	29.18	29.28	0.22	0.32	D
31	29.09	29.27	29.11	29.23	29.18	0.12	0.01	
35	29.19	29.12	29.17	29.17	29.16	0.10	0.01	
24	29.28	29.18	29.01	29.08	29.14	0.08	0.19	
33	29.25	29.25	29.00	29.00	29.13	0.07	0.25	
			[GRAND MEAN]=		29.06	A= 1.77	C= 0.38	
43	29.04	29.01	28.99	28.93	28.99	0.07	0.07	
30	28.84	28.90	29.11	28.68	28.88	0.18	0.02	
22	28.83	28.76	29.09	28.75	28.86	0.20	0.12	
39	28.72	28.76	28.77	28.78	28.76	0.30	0.04	
25	29.03	29.32	28.24	28.24	28.71	0.35	0.94	CD*
44	28.84	28.63	28.70	28.65	28.71	0.35	0.06	
29	28.66	28.65	28.63	28.53	28.62	0.44	0.08	
38	28.65	28.83	27.98	27.98	28.36	0.70	0.76	BCD*
27	28.26	28.29	28.33	28.43	28.33	0.73	0.10	B
20	28.01	28.20	28.15	28.15	28.13	0.93	0.04	B
42	27.00	27.60	26.90	26.80	27.08	1.98	0.45	ABCD
13	26.73	26.91	25.98	26.53	26.54	2.52	0.56	ABCD
32	26.34	26.44	26.40	26.31	26.37	2.69	0.04	AB *
47	25.72	25.82	24.97	24.88	25.35	3.71	0.85	ABCD*

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (0.5)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.3)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 2

Repeatability and reproducibility of per cent ash for sample 21

=(0.30,0.73)

Delete results from laboratory 42

=(0.16,0.47)

CANSPECS
STATISTICAL ANALYSIS OF ASH

30 of 50 reporting sample : 21

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
42	15.10	14.90	13.20	13.20	14.10	1.04	1.80	C *
25	13.50	13.60	15.10	12.80	13.75	0.69	0.40	
32	13.44	13.46	13.53	13.48	13.48	0.42	0.05	
31	13.22	13.63	13.29	13.11	13.31	0.25	0.23	
44	13.43	13.09	13.24	13.27	13.26	0.20	0.01	
48	13.36	13.04	13.48	12.99	13.22	0.16	0.03	
22	13.29	13.20	13.13	13.25	13.22	0.16	0.05	
37	13.05	13.12	13.08	13.25	13.13	0.06	0.08	
15	13.15	13.13	13.11	13.11	13.13	0.06	0.03	
45	13.04	13.16	13.17	13.10	13.12	0.06	0.03	
43	13.18	12.96	13.08	13.21	13.11	0.05	0.07	
10	12.92	13.10	13.18	13.23	13.11	0.05	0.19	
26	13.09	13.02	13.22	13.06	13.10	0.04	0.09	
29	13.18	13.18	12.96	13.07	13.10	0.04	0.17	
35	13.07	12.99	13.18	13.08	13.08	0.02	0.10	
			[GRAND MEAN]=		13.06	A= 1.27 C=	0.63	
19	13.11	13.00	13.07	13.05	13.06	0.00	0.00	
16	13.10	13.00	13.00	12.96	13.02	0.05	0.07	
20	12.88	13.04	13.06	13.02	13.00	0.06	0.08	
3	12.97	12.97	12.97	12.97	12.97	0.09	0.00	
14	13.14	13.10	12.87	12.74	12.96	0.10	0.32	
5	13.01	13.05	12.76	13.01	12.96	0.10	0.15	
12	12.85	12.85	12.95	13.03	12.92	0.14	0.14	
24	12.89	12.81	12.93	12.92	12.89	0.17	0.07	
28	12.81	12.88	12.84	12.94	12.87	0.19	0.04	
18	12.80	12.90	12.90	12.80	12.85	0.21	0.00	
47	12.83	12.85	12.75	12.81	12.81	0.25	0.06	
4	13.28	12.73	12.46	12.65	12.78	0.28	0.45	
36	12.74	12.70	12.67	12.70	12.70	0.36	0.04	
8	12.72	12.60	12.68	12.50	12.63	0.44	0.07	
2	12.00	12.00	12.00	13.00	12.25	0.81	0.50	

CANSPECS
STATISTICAL ANALYSIS OF ASH

30 of 50 reporting sample : 21

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
42	15.10	14.90	13.20	13.20	14.10	1.07	1.80	ABCD*
25	13.50	13.60	15.10	12.80	13.75	0.72	0.40	C
32	13.44	13.46	13.53	13.48	13.48	0.45	0.05	
31	13.22	13.63	13.29	13.11	13.31	0.29	0.23	
44	13.43	13.09	13.24	13.27	13.26	0.23	0.01	
48	13.36	13.04	13.48	12.99	13.22	0.19	0.03	
22	13.29	13.20	13.13	13.25	13.22	0.19	0.05	
37	13.05	13.12	13.08	13.25	13.13	0.10	0.08	
15	13.15	13.13	13.11	13.11	13.13	0.10	0.03	
45	13.04	13.16	13.17	13.10	13.12	0.09	0.03	
43	13.18	12.96	13.08	13.21	13.11	0.08	0.07	
10	12.92	13.10	13.18	13.23	13.11	0.08	0.19	
26	13.09	13.02	13.22	13.06	13.10	0.07	0.09	
29	13.18	13.18	12.96	13.07	13.10	0.07	0.17	
35	13.07	12.99	13.18	13.08	13.08	0.05	0.10	
19	13.11	13.00	13.07	13.05	13.06	0.03	0.00	
			[GRAND MEAN]=		13.03	A= 0.73	C= 0.30	
16	13.10	13.00	13.00	12.96	13.02	0.01	0.07	
20	12.88	13.04	13.06	13.02	13.00	0.03	0.08	
3	12.97	12.97	12.97	12.97	12.97	0.06	0.00	
14	13.14	13.10	12.87	12.74	12.96	0.06	0.32	C
5	13.01	13.05	12.76	13.01	12.96	0.07	0.15	
12	12.85	12.85	12.95	13.03	12.92	0.11	0.14	
24	12.89	12.81	12.93	12.92	12.89	0.14	0.07	
28	12.81	12.88	12.84	12.94	12.87	0.16	0.04	
18	12.80	12.90	12.90	12.80	12.85	0.18	0.00	
47	12.83	12.85	12.75	12.81	12.81	0.22	0.06	
4	13.28	12.73	12.46	12.65	12.78	0.25	0.45	C
36	12.74	12.70	12.67	12.70	12.70	0.32	0.04	
8	12.72	12.60	12.68	12.50	12.63	0.40	0.07	
2	12.00	12.00	12.00	13.00	12.25	0.78	0.50	A C

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (1.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.5)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF ASH

29 of 50 reporting sample : 21

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
25	13.50	13.60	15.10	12.80	13.75	0.72	0.40	C *
32	13.44	13.46	13.53	13.48	13.48	0.45	0.05	
31	13.22	13.63	13.29	13.11	13.31	0.29	0.23	
44	13.43	13.09	13.24	13.27	13.26	0.23	0.01	
48	13.36	13.04	13.48	12.99	13.22	0.19	0.03	
22	13.29	13.20	13.13	13.25	13.22	0.19	0.05	
15	13.15	13.13	13.11	13.11	13.13	0.10	0.03	
37	13.05	13.12	13.08	13.25	13.13	0.10	0.08	
45	13.04	13.16	13.17	13.10	13.12	0.09	0.03	
43	13.18	12.96	13.08	13.21	13.11	0.08	0.07	
10	12.92	13.10	13.18	13.23	13.11	0.08	0.19	
26	13.09	13.02	13.22	13.06	13.10	0.07	0.09	
29	13.18	13.18	12.96	13.07	13.10	0.07	0.17	
35	13.07	12.99	13.18	13.08	13.08	0.05	0.10	
19	13.11	13.00	13.07	13.05	13.06	0.03	0.00	
			[GRAND MEAN]=		13.03	A= 0.73 C=	0.30	
16	13.10	13.00	13.00	12.96	13.02	0.01	0.07	
20	12.88	13.04	13.06	13.02	13.00	0.03	0.08	
3	12.97	12.97	12.97	12.97	12.97	0.06	0.00	
14	13.14	13.10	12.87	12.74	12.96	0.06	0.32	C *
5	13.01	13.05	12.76	13.01	12.96	0.07	0.15	
12	12.85	12.85	12.95	13.03	12.92	0.11	0.14	
24	12.89	12.81	12.93	12.92	12.89	0.14	0.07	
28	12.81	12.88	12.84	12.94	12.87	0.16	0.04	
18	12.80	12.90	12.90	12.80	12.85	0.18	0.00	
47	12.83	12.85	12.75	12.81	12.81	0.22	0.06	
4	13.28	12.73	12.46	12.65	12.78	0.25	0.45	C *
36	12.74	12.70	12.67	12.70	12.70	0.32	0.04	
8	12.72	12.60	12.68	12.50	12.63	0.40	0.07	
2	12.00	12.00	12.00	13.00	12.25	0.78	0.50	A C *

CANSPECS
STATISTICAL ANALYSIS OF ASH

29 of 50 reporting sample : 21

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
25	13.50	13.60	15.10	12.80	13.75	0.71	0.40	A C *
32	13.44	13.46	13.53	13.48	13.48	0.44	0.05	
31	13.22	13.63	13.29	13.11	13.31	0.27	0.23	C
44	13.43	13.09	13.24	13.27	13.26	0.22	0.01	
48	13.36	13.04	13.48	12.99	13.22	0.18	0.03	
22	13.29	13.20	13.13	13.25	13.22	0.18	0.05	
15	13.15	13.13	13.11	13.11	13.13	0.08	0.03	
37	13.05	13.12	13.08	13.25	13.13	0.08	0.08	
45	13.04	13.16	13.17	13.10	13.12	0.08	0.03	
43	13.18	12.96	13.08	13.21	13.11	0.07	0.07	
10	12.92	13.10	13.18	13.23	13.11	0.07	0.19	C
26	13.09	13.02	13.22	13.06	13.10	0.06	0.09	
29	13.18	13.18	12.96	13.07	13.10	0.06	0.17	C
35	13.07	12.99	13.18	13.08	13.08	0.04	0.10	
19	13.11	13.00	13.07	13.05	13.06	0.02	0.00	
			[GRAND MEAN]=		13.04	A= 0.47	C= 0.16	
16	13.10	13.00	13.00	12.96	13.02	0.03	0.07	
20	12.88	13.04	13.06	13.02	13.00	0.04	0.08	
3	12.97	12.97	12.97	12.97	12.97	0.07	0.00	
14	13.14	13.10	12.87	12.74	12.96	0.08	0.32	C *
5	13.01	13.05	12.76	13.01	12.96	0.08	0.15	
12	12.85	12.85	12.95	13.03	12.92	0.12	0.14	
24	12.89	12.81	12.93	12.92	12.89	0.15	0.07	
28	12.81	12.88	12.84	12.94	12.87	0.17	0.04	
18	12.80	12.90	12.90	12.80	12.85	0.19	0.00	
47	12.83	12.85	12.75	12.81	12.81	0.23	0.06	
4	13.28	12.73	12.46	12.65	12.78	0.26	0.45	C *
36	12.74	12.70	12.67	12.70	12.70	0.34	0.04	
8	12.72	12.60	12.68	12.50	12.63	0.42	0.07	
2	12.00	12.00	12.00	13.00	12.25	0.79	0.50	A C *

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (1.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.5)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 3

Repeatability and reproducibility of per cent ash for sample 14

=(0.16,1.52)

Delete results from laboratory 10

=(0.12,0.48)

CANSPECS
STATISTICAL ANALYSIS OF ASH

38 of 50 reporting lab.	run 1	run 2	run 3	run 4	average	difference	sample : 14 range	note
10	24.49	24.47	23.91	23.79	24.17	12.92	0.63	A C *
44	14.74	14.49	15.24	15.05	14.88	3.64	0.53	C *
37	13.53	14.02	13.99	14.00	13.89	2.64	0.22	
3	13.86	13.84	13.79	13.85	13.84	2.59	0.03	
19	11.50	11.60	11.70	11.30	11.52	0.28	0.05	
			[GRAND MEAN]=		11.24 A=	3.87 C=	0.30	
45	10.77	10.83	11.06	11.10	10.94	0.30	0.28	
9	11.10	10.91	10.81	10.87	10.92	0.32	0.16	
34	10.83	10.94	10.85	10.88	10.88	0.37	0.02	
32	10.87	10.85	10.86	10.88	10.87	0.38	0.01	
46	10.89	10.84	10.91	10.80	10.86	0.38	0.01	
35	10.74	10.89	10.82	10.91	10.84	0.40	0.05	
13	10.83	10.85	10.85	10.82	10.84	0.41	0.01	
25	10.80	10.85	10.82	10.83	10.83	0.42	0.00	
48	10.78	10.81	10.83	10.79	10.80	0.44	0.01	
33	10.77	10.79	10.79	10.85	10.80	0.44	0.04	
26	10.77	10.77	10.80	10.82	10.79	0.45	0.04	
4	10.75	10.76	10.79	10.81	10.78	0.47	0.05	
1	10.70	10.77	10.85	10.71	10.76	0.49	0.04	
17	10.65	10.62	10.83	10.85	10.74	0.51	0.20	
24	10.76	10.72	10.73	10.73	10.74	0.51	0.01	
47	10.65	10.66	10.75	10.77	10.71	0.54	0.11	
41	10.70	10.74	10.67	10.71	10.70	0.54	0.03	
42	10.60	10.70	10.70	10.70	10.68	0.57	0.05	
30	10.67	10.65	10.68	10.68	10.67	0.57	0.02	
5	10.32	10.73	10.85	10.71	10.65	0.59	0.26	
27	10.74	10.70	10.55	10.61	10.65	0.59	0.14	
11	10.68	10.58	10.69	10.64	10.65	0.60	0.03	
28	10.56	10.58	10.60	10.64	10.60	0.65	0.05	
14	10.54	10.65	10.52	10.63	10.59	0.66	0.02	
39	10.47	10.61	10.57	10.49	10.53	0.71	0.01	
31	10.47	10.45	10.43	10.46	10.45	0.79	0.01	
18	10.40	10.40	10.40	10.50	10.43	0.82	0.05	
29	10.43	10.39	10.42	10.43	10.42	0.83	0.01	
40	10.00	10.40	10.30	10.94	10.41	0.83	0.42	C *
16	10.40	10.40	10.40	10.38	10.39	0.85	0.01	
22	10.04	10.17	10.17	10.29	10.17	1.08	0.12	
7	8.98	8.98	8.99	8.98	8.98	2.26	0.01	
23	9.02	8.88	8.85	8.93	8.92	2.32	0.06	

CANSPECS
STATISTICAL ANALYSIS OF ASH

38 of 50 reporting — sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
10	24.49	24.47	23.91	23.79	24.17	13.37	0.63	ABCD*
44	14.74	14.49	15.24	15.05	14.88	4.09	0.53	ABCD*
37	13.53	14.02	13.99	14.00	13.89	3.09	0.22	ABC
3	13.86	13.84	13.79	13.85	13.84	3.04	0.03	AB
19	11.50	11.60	11.70	11.30	11.52	0.73	0.05	B
45	10.77	10.83	11.06	11.10	10.94	0.15	0.28	C
9	11.10	10.91	10.81	10.87	10.92	0.13	0.16	C
34	10.83	10.94	10.85	10.88	10.88	0.08	0.02	
32	10.87	10.85	10.86	10.88	10.87	0.07	0.01	
46	10.89	10.84	10.91	10.80	10.86	0.07	0.01	
35	10.74	10.89	10.82	10.91	10.84	0.05	0.05	
13	10.83	10.85	10.85	10.82	10.84	0.04	0.01	
25	10.80	10.85	10.82	10.83	10.83	0.03	0.00	
48	10.78	10.81	10.83	10.79	10.80	0.01	0.01	
33	10.77	10.79	10.79	10.85	10.80	0.01	0.04	
			[GRAND MEAN]=		10.79	A= 1.52	C= 0.16	
26	10.77	10.77	10.80	10.82	10.79	0.00	0.04	
4	10.75	10.76	10.79	10.81	10.78	0.02	0.05	
1	10.70	10.77	10.85	10.71	10.76	0.04	0.04	
17	10.65	10.62	10.83	10.85	10.74	0.06	0.20	C
24	10.76	10.72	10.73	10.73	10.74	0.06	0.01	
47	10.65	10.66	10.75	10.77	10.71	0.09	0.11	
41	10.70	10.74	10.67	10.71	10.70	0.09	0.03	
42	10.60	10.70	10.70	10.70	10.68	0.12	0.05	
30	10.67	10.65	10.68	10.68	10.67	0.12	0.02	
5	10.32	10.73	10.85	10.71	10.65	0.14	0.26	C
27	10.74	10.70	10.55	10.61	10.65	0.14	0.14	
11	10.68	10.58	10.69	10.64	10.65	0.15	0.03	
28	10.56	10.58	10.60	10.64	10.60	0.20	0.05	
14	10.54	10.65	10.52	10.63	10.59	0.21	0.02	
39	10.47	10.61	10.57	10.49	10.53	0.26	0.01	
31	10.47	10.45	10.43	10.46	10.45	0.34	0.01	
18	10.40	10.40	10.40	10.50	10.43	0.37	0.05	
29	10.43	10.39	10.42	10.43	10.42	0.38	0.01	
40	10.00	10.40	10.30	10.94	10.41	0.38	0.42	CD*
16	10.40	10.40	10.40	10.38	10.39	0.40	0.01	
22	10.04	10.17	10.17	10.29	10.17	0.63	0.12	B
7	8.98	8.98	8.99	8.98	8.98	1.81	0.01	AB
23	9.02	8.88	8.85	8.93	8.92	1.87	0.06	AB

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (0.5)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.3)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF ASH

37 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
44	14.74	14.49	15.24	15.05	14.88	3.99	0.53	A C *
37	13.53	14.02	13.99	14.00	13.89	2.99	0.22	A *
3	13.86	13.84	13.79	13.85	13.84	2.94	0.03	A *
19	11.50	11.60	11.70	11.30	11.52	0.63	0.05	
45	10.77	10.83	11.06	11.10	10.94	0.05	0.28	C *
9	11.10	10.91	10.81	10.87	10.92	0.03	0.16	
			[GRAND MEAN]=		10.89	A= 1.83	C= 0.25	
34	10.83	10.94	10.85	10.88	10.88	0.02	0.02	
32	10.87	10.85	10.86	10.88	10.87	0.03	0.01	
46	10.89	10.84	10.91	10.80	10.86	0.03	0.01	
35	10.74	10.89	10.82	10.91	10.84	0.05	0.05	
13	10.83	10.85	10.85	10.82	10.84	0.06	0.01	
25	10.80	10.85	10.82	10.83	10.83	0.07	0.00	
48	10.78	10.81	10.83	10.79	10.80	0.09	0.01	
33	10.77	10.79	10.79	10.85	10.80	0.09	0.04	
26	10.77	10.77	10.80	10.82	10.79	0.10	0.04	
4	10.75	10.76	10.79	10.81	10.78	0.12	0.05	
1	10.70	10.77	10.85	10.71	10.76	0.14	0.04	
17	10.65	10.62	10.83	10.85	10.74	0.16	0.20	
24	10.76	10.72	10.73	10.73	10.74	.16	0.01	
47	10.65	10.66	10.75	10.77	10.71	0.19	0.11	
41	10.70	10.74	10.67	10.71	10.70	0.19	0.03	
42	10.60	10.70	10.70	10.70	10.68	0.22	0.05	
30	10.67	10.65	10.68	10.68	10.67	0.22	0.02	
5	10.32	10.73	10.85	10.71	10.65	0.24	0.26	C *
27	10.74	10.70	10.55	10.61	10.65	0.24	0.14	
11	10.68	10.58	10.69	10.64	10.65	0.25	0.03	
28	10.56	10.58	10.60	10.64	10.60	0.30	0.05	
14	10.54	10.65	10.52	10.63	10.59	0.31	0.02	
39	10.47	10.61	10.57	10.49	10.53	0.36	0.01	
31	10.47	10.45	10.43	10.46	10.45	0.44	0.01	
18	10.40	10.40	10.40	10.50	10.43	0.47	0.05	
29	10.43	10.39	10.42	10.43	10.42	0.48	0.01	
40	10.00	10.40	10.30	10.94	10.41	0.48	0.42	C *
16	10.40	10.40	10.40	10.38	10.39	0.50	0.01	
22	10.04	10.17	10.17	10.29	10.17	0.73	0.12	
7	8.98	8.98	8.99	8.98	8.98	1.91	0.01	A *
23	9.02	8.88	8.85	8.93	8.92	1.97	0.06	A *

CANSPECS
STATISTICAL ANALYSIS OF ASH

37 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
44	14.74	14.49	15.24	15.05	14.88	4.17	0.53	ABCD*
37	13.53	14.02	13.99	14.00	13.89	3.18	0.22	ABC *
3	13.86	13.84	13.79	13.85	13.84	3.13	0.03	AB *
19	11.50	11.60	11.70	11.30	11.52	0.82	0.05	AB
45	10.77	10.83	11.06	11.10	10.94	0.23	0.28	C *
9	11.10	10.91	10.81	10.87	10.92	0.21	0.16	C
34	10.83	10.94	10.85	10.88	10.88	0.17	0.02	
32	10.87	10.85	10.86	10.88	10.87	0.16	0.01	
46	10.89	10.84	10.91	10.80	10.86	0.15	0.01	
35	10.74	10.89	10.82	10.91	10.84	0.13	0.05	
13	10.83	10.85	10.85	10.82	10.84	0.13	0.01	
25	10.80	10.85	10.82	10.83	10.83	0.12	0.00	
48	10.78	10.81	10.83	10.79	10.80	0.09	0.01	
33	10.77	10.79	10.79	10.85	10.80	0.09	0.04	
26	10.77	10.77	10.80	10.82	10.79	0.08	0.04	
4	10.75	10.76	10.79	10.81	10.78	0.07	0.05	
1	10.70	10.77	10.85	10.71	10.76	0.05	0.04	
17	10.65	10.62	10.83	10.85	10.74	0.03	0.20	C
24	10.76	10.72	10.73	10.73	10.74	0.03	0.01	
			[GRAND MEAN]=		10.71	A= 0.48	C= 0.12	
47	10.65	10.66	10.75	10.77	10.71	0.00	0.11	
41	10.70	10.74	10.67	10.71	10.70	0.00	0.03	
42	10.60	10.70	10.70	10.70	10.68	0.03	0.05	
30	10.67	10.65	10.68	10.68	10.67	0.04	0.02	
5	10.32	10.73	10.85	10.71	10.65	0.06	0.26	C *
27	10.74	10.70	10.55	10.61	10.65	0.06	0.14	C
11	10.68	10.58	10.69	10.64	10.65	0.06	0.03	
28	10.56	10.58	10.60	10.64	10.60	0.11	0.05	
14	10.54	10.65	10.52	10.63	10.59	0.12	0.02	
39	10.47	10.61	10.57	10.49	10.53	0.17	0.01	
31	10.47	10.45	10.43	10.46	10.45	0.26	0.01	
18	10.40	10.40	10.40	10.50	10.43	0.28	0.05	
29	10.43	10.39	10.42	10.43	10.42	0.29	0.01	
40	10.00	10.40	10.30	10.94	10.41	0.30	0.42	CD*
16	10.40	10.40	10.40	10.38	10.39	0.31	0.01	
22	10.04	10.17	10.17	10.29	10.17	0.54	0.12	ABC
7	8.98	8.98	8.99	8.98	8.98	1.73	0.01	AB *
23	9.02	8.88	8.85	8.93	8.92	1.79	0.06	AB *

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (0.5)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.3)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 4

Repeatability and reproducibility of per cent volatile matter for sample 11

=(0.78,10.60)

Delete results from laboratories 9 and 35

=(0.42,6.01)

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

35 of 50 reporting sample : 11

lab.	run 1	run 2	run 3	run 4	average	difference	range	note	
9	68.55	71.86	72.56	73.83	71.70	34.46	2.99	A	*
22	57.20	57.15	57.43	57.20	57.25	20.01	0.14	A	*
48	56.97	56.21	57.44	57.28	56.97	19.74	0.77	A	*
32	56.88	56.66	56.50	56.22	56.57	19.33	0.41		
30	50.55	50.62	51.02	51.30	50.87	13.63	0.58		
17	45.43	45.43	45.28	45.25	45.35	8.11	0.17		
45	41.97	43.78	42.74	41.83	42.58	5.34	0.59		
39	42.03	42.07	40.01	41.53	41.41	4.17	1.28		
33	35.83	37.20	38.30	38.16	37.37	0.13	1.71		
			[GRAND MEAN]=		37.24	A= 19.44	C= 5.66		
3	35.77	33.53	35.80	34.47	34.89	2.35	0.48		
31	34.46	33.65	33.67	34.57	34.09	3.15	0.06		
41	33.77	33.91	34.17	33.98	33.96	3.28	0.23		
8	33.85	33.80	33.75	33.80	33.80	3.44	0.05		
24	33.91	33.98	33.60	33.66	33.79	3.45	0.32		
16	33.78	33.82	33.10	33.30	33.50	3.74	0.60		
38	33.20	33.60	33.30	33.50	33.40	3.84	0.00		
18	33.60	33.20	33.00	33.40	33.30	3.94	0.20		
29	33.26	33.15	33.64	33.14	33.30	3.94	0.18		
14	33.61	32.98	33.93	32.53	33.26	3.98	0.07		
2	33.20	33.15	33.27	33.21	33.21	4.03	0.06		
1	33.10	33.25	33.05	33.15	33.14	4.10	0.07		
44	32.86	33.84	32.60	33.23	33.13	4.11	0.44		
25	33.30	32.96	33.00	33.14	33.10	4.14	0.06		
28	32.56	32.53	32.56	32.62	32.57	4.67	0.04		
26	32.15	32.73	32.87	32.28	32.51	4.73	0.13		
43	32.46	32.23	32.55	32.33	32.39	4.85	0.09		
13	32.36	32.48	32.21	32.16	32.30	4.94	0.24		
47	32.14	32.03	32.11	32.20	32.12	5.12	0.07		
11	32.19	31.80	32.75	31.72	32.11	5.12	0.24		
34	32.09	31.96	31.99	32.16	32.05	5.19	0.05		
21	31.88	32.12	31.99	32.13	32.03	5.21	0.06		
37	31.47	31.74	31.73	31.45	31.60	5.64	0.01		
5	30.78	30.66	30.69	30.66	30.70	6.54	0.05		
46	30.01	30.24	30.21	30.67	30.28	6.96	0.31		
35	32.51	32.55	13.12	12.96	22.78	14.45	19.49	C	*

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

35 of 50 reporting sample : 11

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
9	68.55	71.86	72.56	73.83	71.70	36.39	2.99	ABCD*
22	57.20	57.15	57.43	57.20	57.25	21.93	0.14	AB *
48	56.97	56.21	57.44	57.28	56.97	21.66	0.77	AB D*
32	56.88	56.66	56.50	56.22	56.57	21.25	0.41	AB
30	50.55	50.62	51.02	51.30	50.87	15.56	0.58	AB
17	45.43	45.43	45.28	45.25	45.35	10.04	0.17	B
45	41.97	43.78	42.74	41.83	42.58	7.27	0.59	B
39	42.03	42.07	40.01	41.53	41.41	6.10	1.28	BCD
33	35.83	37.20	38.30	38.16	37.37	2.06	1.71	BCD
			[GRAND MEAN]=		35.31	A= 10.60	C= 0.78	
3	35.77	33.53	35.80	34.47	34.89	0.42	0.48	
31	34.46	33.65	33.67	34.57	34.09	1.22	0.06	
41	33.77	33.91	34.17	33.98	33.96	1.35	0.23	
8	33.85	33.80	33.75	33.80	33.80	1.51	0.05	B
24	33.91	33.98	33.60	33.66	33.79	1.52	0.32	B
16	33.78	33.82	33.10	33.30	33.50	1.81	0.60	B
38	33.20	33.60	33.30	33.50	33.40	1.91	0.00	B
18	33.60	33.20	33.00	33.40	33.30	2.01	0.20	B
29	33.26	33.15	33.64	33.14	33.30	2.01	0.18	B
14	33.61	32.98	33.93	32.53	33.26	2.05	0.07	B
2	33.20	33.15	33.27	33.21	33.21	2.10	0.06	B
1	33.10	33.25	33.05	33.15	33.14	2.17	0.07	B
44	32.86	33.84	32.60	33.23	33.13	2.18	0.44	B
25	33.30	32.96	33.00	33.14	33.10	2.21	0.06	B
28	32.56	32.53	32.56	32.62	32.57	2.74	0.04	B
26	32.15	32.73	32.87	32.28	32.51	2.80	0.13	B
43	32.46	32.23	32.55	32.33	32.39	2.92	0.09	B
13	32.36	32.48	32.21	32.16	32.30	3.01	0.24	B
47	32.14	32.03	32.11	32.20	32.12	3.19	0.07	B
11	32.19	31.80	32.75	31.72	32.11	3.20	0.24	B
34	32.09	31.96	31.99	32.16	32.05	3.26	0.05	B
21	31.88	32.12	31.99	32.13	32.03	3.28	0.06	B
37	31.47	31.74	31.73	31.45	31.60	3.71	0.01	B
5	30.78	30.66	30.69	30.66	30.70	4.61	0.05	B
46	30.01	30.24	30.21	30.67	30.28	5.03	0.31	B
35	32.51	32.55	13.12	12.96	22.78	12.53	19.49	ABCD*

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (1.4)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.7)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

33 of 50 reporting sample : 11

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
22	57.20	57.15	57.43	57.20	57.25	20.61	0.14	A *
48	56.97	56.21	57.44	57.28	56.97	20.34	0.77	A *
32	56.88	56.66	56.50	56.22	56.57	19.93	0.41	A *
30	50.55	50.62	51.02	51.30	50.87	14.24	0.58	A *
17	45.43	45.43	45.28	45.25	45.35	8.71	0.17	
45	41.97	43.78	42.74	41.83	42.58	5.95	0.59	
39	42.03	42.07	40.01	41.53	41.41	4.78	1.28	C *
33	35.83	37.20	38.30	38.16	37.37	0.74	1.71	C *
			[GRAND MEAN]=		36.63	A= 13.44	C= 0.79	
3	35.77	33.53	35.80	34.47	34.89	1.74	0.48	
31	34.46	33.65	33.67	34.57	34.09	2.55	0.06	
41	33.77	33.91	34.17	33.98	33.96	2.68	0.23	
8	33.85	33.80	33.75	33.80	33.80	2.83	0.05	
24	33.91	33.98	33.60	33.66	33.79	2.85	0.32	
16	33.78	33.82	33.10	33.30	33.50	3.13	0.60	
38	33.20	33.60	33.30	33.50	33.40	3.23	0.00	
18	33.60	33.20	33.00	33.40	33.30	3.33	0.20	
29	33.26	33.15	33.64	33.14	33.30	3.34	0.18	
14	33.61	32.98	33.93	32.53	33.26	3.37	0.07	
2	33.20	33.15	33.27	33.21	33.21	3.43	0.06	
1	33.10	33.25	33.05	33.15	33.14	3.50	0.07	
44	32.86	33.84	32.60	33.23	33.13	3.50	0.44	
25	33.30	32.96	33.00	33.14	33.10	3.53	0.06	
28	32.56	32.53	32.56	32.62	32.57	4.07	0.04	
26	32.15	32.73	32.87	32.28	32.51	4.13	0.13	
43	32.46	32.23	32.55	32.33	32.39	4.24	0.09	
13	32.36	32.48	32.21	32.16	32.30	4.33	0.24	
47	32.14	32.03	32.11	32.20	32.12	4.51	0.07	
11	32.19	31.80	32.75	31.72	32.11	4.52	0.24	
34	32.09	31.96	31.99	32.16	32.05	4.58	0.05	
21	31.88	32.12	31.99	32.13	32.03	4.60	0.06	
37	31.47	31.74	31.73	31.45	31.60	5.04	0.01	
5	30.78	30.66	30.69	30.66	30.70	5.94	0.05	
46	30.01	30.24	30.21	30.67	30.28	6.35	0.31	

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

33 of 50 reporting sample : 11

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
22	57.20	57.15	57.43	57.20	57.25	23.60	0.14	AB *
48	56.97	56.21	57.44	57.28	56.97	23.33	0.77	ABC *
32	56.88	56.66	56.50	56.22	56.57	22.92	0.41	AB *
30	50.55	50.62	51.02	51.30	50.87	17.23	0.58	ABC *
17	45.43	45.43	45.28	45.25	45.35	11.70	0.17	AB
45	41.97	43.78	42.74	41.83	42.58	8.93	0.59	ABC
39	42.03	42.07	40.01	41.53	41.41	7.76	1.28	ABCD*
33	35.83	37.20	38.30	38.16	37.37	3.73	1.71	BCD*
3	35.77	33.53	35.80	34.47	34.89	1.25	0.48	C
31	34.46	33.65	33.67	34.57	34.09	0.44	0.06	
41	33.77	33.91	34.17	33.98	33.96	0.31	0.23	
8	33.85	33.80	33.75	33.80	33.80	0.15	0.05	
24	33.91	33.98	33.60	33.66	33.79	0.14	0.32	
			[GRAND MEAN]=		33.65	A= 6.01	C= 0.42	
16	33.78	33.82	33.10	33.30	33.50	0.15	0.60	C
38	33.20	33.60	33.30	33.50	33.40	0.25	0.00	
18	33.60	33.20	33.00	33.40	33.30	0.35	0.20	
29	33.26	33.15	33.64	33.14	33.30	0.35	0.18	
14	33.61	32.98	33.93	32.53	33.26	0.38	0.07	
2	33.20	33.15	33.27	33.21	33.21	0.44	0.06	
1	33.10	33.25	33.05	33.15	33.14	0.51	0.07	
44	32.86	33.84	32.60	33.23	33.13	0.51	0.44	C
25	33.30	32.96	33.00	33.14	33.10	0.55	0.06	
28	32.56	32.53	32.56	32.62	32.57	1.08	0.04	
26	32.15	32.73	32.87	32.28	32.51	1.14	0.13	
43	32.46	32.23	32.55	32.33	32.39	1.25	0.09	
13	32.36	32.48	32.21	32.16	32.30	1.34	0.24	
47	32.14	32.03	32.11	32.20	32.12	1.53	0.07	
11	32.19	31.80	32.75	31.72	32.11	1.53	0.24	
34	32.09	31.96	31.99	32.16	32.05	1.60	0.05	
21	31.88	32.12	31.99	32.13	32.03	1.62	0.06	
37	31.47	31.74	31.73	31.45	31.60	2.05	0.01	B
5	30.78	30.66	30.69	30.66	30.70	2.95	0.05	B
46	30.01	30.24	30.21	30.67	30.28	3.36	0.31	B

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (1.4)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (0.7)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 5

Repeatability and reproducibility of per cent volatile matter for sample 4

=(0.33,21.44)

Delete results from laboratory 19

=(0.28,13.35)

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

27 of 50 reporting lab.	run 1	run 2	run 3	run 4	average	difference	sample : 4 range	note
19	85.20	86.10	86.20	83.20	85.18	48.86	0.95	A C *
36	58.84	58.75	59.08	58.90	58.89	22.58	0.19	
27	59.00	58.65	58.80	58.60	58.76	22.45	0.13	
17	58.00	57.50	57.95	58.07	57.88	21.57	0.26	
22	50.74	50.71	50.69	50.55	50.67	14.36	0.11	
30	43.46	43.71	43.89	43.33	43.60	7.29	0.03	
10	38.43	37.92	38.71	37.99	38.26	1.95	0.18	
			[GRAND MEAN]=		36.31	A= 26.61 C=	0.51	
32	36.22	36.09	35.99	35.91	36.05	0.26	0.21	
23	31.46	31.04	31.31	31.40	31.30	5.01	0.10	
33	31.00	31.31	31.31	31.40	31.26	5.06	0.20	
35	30.57	30.80	30.68	30.69	30.69	5.63	0.00	
4	30.34	30.43	30.44	29.98	30.30	6.01	0.18	
8	30.51	30.48	29.90	29.95	30.21	6.10	0.57	C *
31	29.86	29.61	29.57	29.49	29.63	6.68	0.21	
34	29.00	28.79	29.55	29.35	29.17	7.14	0.55	C *
25	28.94	28.86	28.82	28.88	28.88	7.44	0.05	
2	28.91	28.87	28.66	28.43	28.72	7.59	0.35	
6	29.29	28.19	28.67	28.11	28.57	7.75	0.35	
37	28.77	28.46	28.57	28.46	28.57	7.75	0.10	
21	28.35	28.55	28.57	28.62	28.52	7.79	0.14	
18	28.43	28.42	28.40	28.50	28.44	7.87	0.02	
38	28.00	28.10	28.20	28.30	28.15	8.16	0.20	
29	28.07	28.10	27.92	28.07	28.04	8.27	0.09	
24	27.83	27.92	27.73	27.97	27.86	8.45	0.03	
1	27.38	27.81	27.94	28.25	27.84	8.47	0.50	
28	27.65	27.71	27.55	27.63	27.64	8.68	0.09	
11	27.26	27.26	27.23	27.54	27.32	8.99	0.12	

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

27 of 50 reporting sample : 4

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
19	85.20	86.10	86.20	83.20	85.18	50.35	0.95	ABC *
36	58.84	58.75	59.08	58.90	58.89	24.07	0.19	AB
27	59.00	58.65	58.80	58.60	58.76	23.94	0.13	AB
17	58.00	57.50	57.95	58.07	57.88	23.05	0.26	AB
22	50.74	50.71	50.69	50.55	50.67	15.85	0.11	B
30	43.46	43.71	43.89	43.33	43.60	8.77	0.03	B
10	38.43	37.92	38.71	37.99	38.26	3.44	0.18	B
32	36.22	36.09	35.99	35.91	36.05	1.23	0.21	
			[GRAND MEAN]=		34.83	A= 21.44	C= 0.33	
23	31.46	31.04	31.31	31.40	31.30	3.52	0.10	B
33	31.00	31.31	31.31	31.40	31.26	3.57	0.20	B
35	30.57	30.80	30.68	30.69	30.69	4.14	0.00	B
4	30.34	30.43	30.44	29.98	30.30	4.53	0.18	B
8	30.51	30.48	29.90	29.95	30.21	4.62	0.57	BC *
31	29.86	29.61	29.57	29.49	29.63	5.19	0.21	B
34	29.00	28.79	29.55	29.35	29.17	5.65	0.55	BC *
25	28.94	28.86	28.82	28.88	28.88	5.95	0.05	B
2	28.91	28.87	28.66	28.43	28.72	6.11	0.35	BC
6	29.29	28.19	28.67	28.11	28.57	6.26	0.35	BC
37	28.77	28.46	28.57	28.46	28.57	6.26	0.10	B
21	28.35	28.55	28.57	28.62	28.52	6.30	0.14	B
18	28.43	28.42	28.40	28.50	28.44	6.39	0.02	B
38	28.00	28.10	28.20	28.30	28.15	6.68	0.20	B
29	28.07	28.10	27.92	28.07	28.04	6.79	0.09	B
24	27.83	27.92	27.73	27.97	27.86	6.96	0.03	B
1	27.38	27.81	27.94	28.25	27.84	6.98	0.50	BC
28	27.65	27.71	27.55	27.63	27.64	7.19	0.09	B
11	27.26	27.26	27.23	27.54	27.32	7.50	0.12	B

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (2.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (1.0)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

26 of 50 reporting lab.	run 1	run 2	run 3	run 4	average	difference	sample : 4 range	note
36	58.84	58.75	59.08	58.90	58.89	24.46	0.19	A *
27	59.00	58.65	58.80	58.60	58.76	24.33	0.13	A *
17	58.00	57.50	57.95	58.07	57.88	23.45	0.26	A *
22	50.74	50.71	50.69	50.55	50.67	16.24	0.11	
30	43.46	43.71	43.89	43.33	43.60	9.17	0.03	
10	38.43	37.92	38.71	37.99	38.26	3.83	0.18	
32	36.22	36.09	35.99	35.91	36.05	1.6?	0.21	
			[GRAND MEAN]=		34.43	A= 19.95 C=	0.41	
23	31.46	31.04	31.31	31.40	31.30	3.13	0.10	
33	31.00	31.31	31.31	31.40	31.26	3.18	0.20	
35	30.57	30.80	30.68	30.69	30.69	3.75	0.00	
4	30.34	30.43	30.44	29.98	30.30	4.13	0.18	
8	30.51	30.48	29.90	29.95	30.21	4.22	0.57	C *
31	29.86	29.61	29.57	29.49	29.63	4.80	0.21	
34	29.00	28.79	29.55	29.35	29.17	5.26	0.55	C *
25	28.94	28.86	28.82	28.88	28.88	5.56	0.05	
2	28.91	28.87	28.66	28.43	28.72	5.71	0.35	
6	29.29	28.19	28.67	28.11	28.57	5.87	0.35	
37	28.77	28.46	28.57	28.46	28.57	5.87	0.10	
21	28.35	28.55	28.57	28.62	28.52	5.91	0.14	
18	28.43	28.42	28.40	28.50	28.44	5.99	0.02	
38	28.00	28.10	28.20	28.30	28.15	6.28	0.20	
29	28.07	28.10	27.92	28.07	28.04	6.39	0.09	
24	27.83	27.92	27.73	27.97	27.86	6.57	0.03	
1	27.38	27.81	27.94	28.25	27.84	6.59	0.50	C *
28	27.65	27.71	27.55	27.63	27.64	6.80	0.09	
11	27.26	27.26	27.23	27.54	27.32	7.11	0.12	

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

26 of 50 reporting sample : 4

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
36	58.84	58.75	59.08	58.90	58.89	27.27	0.19	AB *
27	59.00	58.65	58.80	58.60	58.76	27.14	0.13	AB *
17	58.00	57.50	57.95	58.07	57.88	26.26	0.26	AB *
22	50.74	50.71	50.69	50.55	50.67	19.05	0.11	AB
30	43.46	43.71	43.89	43.33	43.60	11.97	0.03	B
10	38.43	37.92	38.71	37.99	38.26	6.64	0.18	B
32	36.22	36.09	35.99	35.91	36.05	4.43	0.21	B
			[GRAND MEAN]=		31.62	A= 13.35 C=	0.28	
23	31.46	31.04	31.31	31.40	31.30	0.32	0.10	
33	31.00	31.31	31.31	31.40	31.26	0.37	0.20	
35	30.57	30.80	30.68	30.69	30.69	0.94	0.00	
4	30.34	30.43	30.44	29.98	30.30	1.33	0.18	
8	30.51	30.48	29.90	29.95	30.21	1.41	0.57	C *
31	29.86	29.61	29.57	29.49	29.63	1.99	0.21	
34	29.00	28.79	29.55	29.35	29.17	2.45	0.55	BC *
25	28.94	28.86	28.82	28.88	28.88	2.75	0.05	B
2	28.91	28.87	28.66	28.43	28.72	2.91	0.35	BC
6	29.29	28.19	28.67	28.11	28.57	3.06	0.35	BC
37	28.77	28.46	28.57	28.46	28.57	3.06	0.10	B
21	28.35	28.55	28.57	28.62	28.52	3.10	0.14	B
18	28.43	28.42	28.40	28.50	28.44	3.19	0.02	B
38	28.00	28.10	28.20	28.30	28.15	3.47	0.20	B
29	28.07	28.10	27.92	28.07	28.04	3.58	0.09	B
24	27.83	27.92	27.73	27.97	27.86	3.76	0.03	B
1	27.38	27.81	27.94	28.25	27.84	3.78	0.50	BC *
28	27.65	27.71	27.55	27.63	27.64	3.99	0.09	B
11	27.26	27.26	27.23	27.54	27.32	4.30	0.12	B

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (2.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (1.0)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 6

Repeatability and reproducibility of per cent volatile matter for sample 14

=(0.44,12.51)

Delete results from laboratory 3

=(0.44,11.45)

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

35 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
3	58.00	58.30	59.30	58.89	58.62	21.93	0.94	AB *
17	53.91	54.02	53.00	53.88	53.70	17.01	0.52	AB *
32	53.15	53.11	53.25	53.32	53.21	16.52	0.15	AB *
45	49.97	50.76	51.60	51.85	51.05	14.36	1.36	BCD*
9	51.10	50.63	51.03	51.16	50.98	14.29	0.23	B
16	50.10	49.90	49.80	49.70	49.88	13.19	0.25	B
10	48.69	48.43	48.57	48.24	48.48	11.79	0.16	B
41	47.02	47.02	46.97	46.97	47.00	10.31	0.05	B
25	44.30	45.17	42.60	42.31	43.60	6.91	2.28	BCD*
14	41.00	41.10	40.76	41.51	41.09	4.40	0.08	B
37	39.89	40.57	41.07	39.97	40.38	3.69	0.29	B
48	38.89	38.75	39.04	39.07	38.94	2.25	0.24	B
			[GRAND MEAN]=		36.69	A= 15.19 C=	0.98	
24	33.00	33.45	33.29	33.06	33.20	3.49	0.05	B
5	32.05	33.22	32.73	33.04	32.76	3.93	0.25	B
4	32.52	32.59	32.74	32.81	32.67	4.02	0.22	B
22	32.09	32.16	32.49	32.66	32.35	4.34	0.45	B
1	32.47	31.83	31.34	32.85	32.12	4.57	0.06	B
30	32.09	32.11	32.10	31.78	32.02	4.67	0.16	B
33	30.87	32.65	31.40	31.90	31.71	4.98	0.11	B
28	31.45	31.52	31.84	31.93	31.69	5.00	0.40	B
26	31.59	31.47	31.66	31.45	31.54	5.15	0.02	B
18	30.60	30.80	32.00	32.10	31.38	5.31	1.35	BCD*
35	31.50	31.37	30.92	31.42	31.30	5.39	0.27	B
46	30.95	30.99	31.25	30.59	30.95	5.74	0.05	B
29	30.56	31.00	30.73	30.59	30.72	5.97	0.12	B
13	30.35	30.21	30.35	30.46	30.34	6.35	0.12	B
6	29.65	29.77	30.05	30.08	29.89	6.80	0.35	B
23	30.14	29.74	29.70	29.83	29.85	6.84	0.18	B
31	29.94	29.46	30.26	29.39	29.76	6.93	0.12	B
27	29.11	29.40	30.97	29.23	29.68	7.01	0.84	B
44	28.89	28.97	29.49	28.92	29.07	7.62	0.27	B
39	28.94	28.91	28.95	29.09	28.97	7.72	0.09	B
34	28.68	28.70	28.90	28.73	28.75	7.94	0.12	B
11	28.68	28.45	28.81	28.59	28.63	8.06	0.13	B
47	27.77	27.73	27.92	27.91	27.83	8.86	0.16	B

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

35 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
3	58.00	58.30	59.30	58.89	58.62	24.40	0.94	ABC *
17	53.91	54.02	53.00	53.88	53.70	19.48	0.52	ABC *
32	53.15	53.11	53.25	53.32	53.21	18.98	0.15	AB *
45	49.97	50.76	51.60	51.85	51.05	16.82	1.36	ABCD*
9	51.10	50.63	51.03	51.16	50.98	16.75	0.23	AB
16	50.10	49.90	49.80	49.70	49.88	15.65	0.25	AB
10	48.69	48.43	48.57	48.24	48.48	14.26	0.16	AB
41	47.02	47.02	46.97	46.97	47.00	12.77	0.05	AB
25	44.30	45.17	42.60	42.31	43.60	9.37	2.28	BCD*
14	41.00	41.10	40.76	41.51	41.09	6.87	0.08	B
37	39.89	40.57	41.07	39.97	40.38	6.15	0.29	B
48	38.89	38.75	39.04	39.07	38.94	4.71	0.24	B
			[GRAND MEAN]=		34.23	A= 12.51	C= 0.44	
24	33.00	33.45	33.29	33.06	33.20	1.03	0.05	B
5	32.05	33.22	32.73	33.04	32.76	1.47	0.25	B
4	32.52	32.59	32.74	32.81	32.67	1.56	0.22	B
22	32.09	32.16	32.49	32.66	32.35	1.88	0.45	BC
1	32.47	31.83	31.34	32.85	32.12	2.10	0.06	B
30	32.09	32.11	32.10	31.78	32.02	2.21	0.16	B
33	30.87	32.65	31.40	31.90	31.71	2.52	0.11	B
28	31.45	31.52	31.84	31.93	31.69	2.54	0.40	B
26	31.59	31.47	31.66	31.45	31.54	2.68	0.02	B
18	30.60	30.80	32.00	32.10	31.38	2.85	1.35	BCD*
35	31.50	31.37	30.92	31.42	31.30	2.92	0.27	B
46	30.95	30.99	31.25	30.59	30.95	3.28	0.05	B
29	30.56	31.00	30.73	30.59	30.72	3.51	0.12	B
13	30.35	30.21	30.35	30.46	30.34	3.88	0.12	B
6	29.65	29.77	30.05	30.08	29.89	4.34	0.35	B
23	30.14	29.74	29.70	29.83	29.85	4.37	0.18	B
31	29.94	29.46	30.26	29.39	29.76	4.46	0.12	B
27	29.11	29.40	30.97	29.23	29.68	4.55	0.84	BC
44	28.89	28.97	29.49	28.92	29.07	5.16	0.27	B
39	28.94	28.91	28.95	29.09	28.97	5.25	0.09	B
34	28.68	28.70	28.90	28.73	28.75	5.47	0.12	B
11	28.68	28.45	28.81	28.59	28.63	5.59	0.13	B
47	27.77	27.73	27.92	27.91	27.83	6.39	0.16	B

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (2.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (1.0)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

34 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
17	53.91	54.02	53.00	53.88	53.70	17.66	0.52	AB *
32	53.15	53.11	53.25	53.32	53.21	17.16	0.15	AB *
45	49.97	50.76	51.60	51.85	51.05	15.00	1.36	ABCD*
9	51.10	50.63	51.03	51.16	50.98	14.94	0.23	AB *
16	50.10	49.90	49.80	49.70	49.88	13.83	0.25	B
10	48.69	48.43	48.57	48.24	48.48	12.44	0.16	B
41	47.02	47.02	46.97	46.97	47.00	10.95	0.05	B
25	44.30	45.17	42.60	42.31	43.60	7.55	2.28	BCD*
14	41.00	41.10	40.76	41.51	41.09	5.05	0.08	B
37	39.89	40.57	41.07	39.97	40.38	4.33	0.29	B
48	38.89	38.75	39.04	39.07	38.94	2.89	0.24	B
			[GRAND MEAN]=		36.04	A= 14.21	C= 0.96	
24	33.00	33.45	33.29	33.06	33.20	2.84	0.05	B
5	32.05	33.22	32.73	33.04	32.76	3.28	0.25	B
4	32.52	32.59	32.74	32.81	32.67	3.38	0.22	B
22	32.09	32.16	32.49	32.66	32.35	3.69	0.45	B
1	32.47	31.83	31.34	32.85	32.12	3.92	0.06	B
30	32.09	32.11	32.10	31.78	32.02	4.02	0.16	B
33	30.87	32.65	31.40	31.90	31.71	4.34	0.11	B
28	31.45	31.52	31.84	31.93	31.69	4.36	0.40	B
26	31.59	31.47	31.66	31.45	31.54	4.50	0.02	B
18	30.60	30.80	32.00	32.10	31.38	4.67	1.35	BCD*
35	31.50	31.37	30.92	31.42	31.30	4.74	0.27	B
46	30.95	30.99	31.25	30.59	30.95	5.10	0.05	B
29	30.56	31.00	30.73	30.59	30.72	5.32	0.12	B
13	30.35	30.21	30.35	30.46	30.34	5.70	0.12	B
6	29.65	29.77	30.05	30.08	29.89	6.16	0.35	B
23	30.14	29.74	29.70	29.83	29.85	6.19	0.18	B
31	29.94	29.46	30.26	29.39	29.76	6.28	0.12	B
27	29.11	29.40	30.97	29.23	29.68	6.37	0.84	B
44	28.89	28.97	29.49	28.92	29.07	6.98	0.27	B
39	28.94	28.91	28.95	29.09	28.97	7.07	0.09	B
34	28.68	28.70	28.90	28.73	28.75	7.29	0.12	B
11	28.68	28.45	28.81	28.59	28.63	7.41	0.13	B
47	27.77	27.73	27.92	27.91	27.83	8.21	0.16	B

CANSPECS
STATISTICAL ANALYSIS OF VOLATILE MATTER

34 of 50 reporting sample : 14

lab.	run 1	run 2	run 3	run 4	average	difference	range	note
17	53.91	54.02	53.00	53.88	53.70	20.08	0.52	ABC *
32	53.15	53.11	53.25	53.32	53.21	19.58	0.15	AB *
45	49.97	50.76	51.60	51.85	51.05	17.42	1.36	ABCD*
9	51.10	50.63	51.03	51.16	50.98	17.35	0.23	AB *
16	50.10	49.90	49.80	49.70	49.88	16.25	0.25	AB
10	48.69	48.43	48.57	48.24	48.48	14.86	0.16	AB
41	47.02	47.02	46.97	46.97	47.00	13.37	0.05	AB
25	44.30	45.17	42.60	42.31	43.60	9.97	2.28	BCD*
14	41.00	41.10	40.76	41.51	41.09	7.47	0.08	B
37	39.89	40.57	41.07	39.97	40.38	6.75	0.29	B
48	38.89	38.75	39.04	39.07	38.94	5.31	0.24	B
			[GRAND MEAN]=		33.63	A= 11.45	C= 0.44	
24	33.00	33.45	33.29	33.06	33.20	0.43	0.05	B
5	32.05	33.22	32.73	33.04	32.76	0.87	0.25	B
4	32.52	32.59	32.74	32.81	32.67	0.96	0.22	B
22	32.09	32.16	32.49	32.66	32.35	1.28	0.45	BC
1	32.47	31.83	31.34	32.85	32.12	1.50	0.06	B
30	32.09	32.11	32.10	31.78	32.02	1.61	0.16	B
33	30.87	32.65	31.40	31.90	31.71	1.92	0.11	B
28	31.45	31.52	31.84	31.93	31.69	1.94	0.40	B
26	31.59	31.47	31.66	31.45	31.54	2.08	0.02	B
18	30.60	30.80	32.00	32.10	31.38	2.25	1.35	BCD*
35	31.50	31.37	30.92	31.42	31.30	2.32	0.27	B
46	30.95	30.99	31.25	30.59	30.95	2.68	0.05	B
29	30.56	31.00	30.73	30.59	30.72	2.91	0.12	B
13	30.35	30.21	30.35	30.46	30.34	3.28	0.12	B
6	29.65	29.77	30.05	30.08	29.89	3.74	0.35	B
23	30.14	29.74	29.70	29.83	29.85	3.77	0.18	B
31	29.94	29.46	30.26	29.39	29.76	3.86	0.12	B
27	29.11	29.40	30.97	29.23	29.68	3.95	0.84	BC
44	28.89	28.97	29.49	28.92	29.07	4.56	0.27	B
39	28.94	28.91	28.95	29.09	28.97	4.65	0.09	B
34	28.68	28.70	28.90	28.73	28.75	4.87	0.12	B
11	28.68	28.45	28.81	28.59	28.63	4.99	0.13	B
47	27.77	27.73	27.92	27.91	27.83	5.79	0.16	B

A > REPRODUCIBILITY CALCULATED BY E-691 :B > ASTM REPRODUCIBILITY (2.0)
C > REPEATABILITY CALCULATED BY E-691 :D > ASTM REPEATABILITY (1.0)
OUTLIERS NOT USED AS CALCULATED BY E-691 FLAGGED BY `*`

APPENDIX 7

Repeatability and reproducibility of volatile matter versus rank number.

Assumption: each coal rank is given an ascending rank number;

Rank	
Name	Number
LIGNITE B	13
LIGNITE A	12
SUBBITUMINOUS C	11
SUBBITUMINOUS B	10
SUBBITUMINOUS A	9
HIGH VOLATILE C BITUMINOUS	8
HIGH VOLATILE B BITUMINOUS	7
HIGH VOLATILE A BITUMINOUS	6
MEDIUM VOLATILE BITUMINOUS	5
LOW VOLATILE BITUMINOUS	4
SEMIANTHRACITE	3
ANTHRACITE	2
META ANTHRACITE	1

Table 3.* Volatile Analysis by ASTM D3175 (with amendments as in discussion)

Coal Rank		Number of	Volatile	Repeatability		Reproducibilty	
Name	Number	Labs.	%	by E691	Accepted	by E691	Accepted
Lignite A	12	27	34.83	0.28	1.0	13.35	2.0
Lignite A	12	35	34.23	0.44	1.0	11.45	2.0
SubBit. C	11	38	35.31	0.42	1.0	6.01	2.0
SubBit. B	10	28	32.42	0.38	0.7	6.11	1.4
SubBit. B	10	30	33.44	0.55	0.7	7.38	1.4
H.V. C Bit.	8	23	36.08	0.35	0.5	5.06	1.0
H.V. C Bit.	8	35	34.93	0.38	0.5	3.58	1.0
H.V. C Bit.	8	35	20.40	0.22	0.5	1.30	1.0
H.V. C Bit.	8	38	32.88	0.42	0.5	2.33	1.0
H.V. B Bit.	7	40	33.44	0.34	0.5	2.98	1.0
H.V. B Bit.	7	35	24.56	0.19	0.5	1.16	1.0
H.V. A Bit.	6	29	34.70	0.29	0.5	2.10	1.0
H.V. A Bit.	6	38	34.51	0.43	0.5	1.91	1.0
H.V. A Bit.	6	33	30.93	0.31	0.5	1.46	1.0
M.V. Bit.	5	24	21.08	0.35	0.5	2.36	1.0
M.V. Bit.	5	30	20.26	0.23	0.5	1.21	1.0
M.V. Bit.	5	35	20.27	0.32	0.5	1.48	1.0
M.V. Bit.	5	39	21.08	0.24	0.5	1.23	1.0
M.V. Bit.	5	38	29.63	0.34	0.5	1.77	1.0
L.V. Bit.	4	24	13.77	0.19	0.5	1.38	1.0
L.V. Bit.	4	29	27.57	0.48	0.5	1.56	1.0

Note: Repeatability and reproducibility are related to coal rank: bituminous 0.5,1.0; subbituminous 0.7,1.4 and lignite 1.0,2.0, respectively

Repeatability and reproducibility of per cent volatile versus rank number

Repeatability

Repeatability = (0.014 +/- 0.017)*(per cent volatile) + (0.24 +/- 0.13)

Coefficient of determination = 0.126

Reproducibility

Reproducibility = (1.19 +/- 0.32)*(per cent volatile) - (4.9 +/- 2.5)

Coefficient of determination = 0.760

APPENDIX 8

Methods quoted as being used in the data presented;

Samples 1 to 18

The Canadian Interlaboratory Round Robin (CIRR)

Samples 19 to 24

CANMET`s Service Program for the Analysis of Characterization Samples (CANSPECS)

for the analysis of:
- Moisture
- Ash
- Volatile matter
- sulphur
- Calorific value (Heat Value)

Definition of symbols:
- * = No Response for this sample
- - = No data for this analysis
- + = ASTM method as referenced in Technical Report
- = Not Stated (usually assumed as no change)
- 1 = Alternate Method 1 (ISO Methods)
- 2 = Alternate Method 2 (alternate ASTM`s usually)
- 3 = Alternate Method 3

Note: Round Robin Sample Exclusions:

CIRR 1	Not enough data for statistically meaningful inclusion.
CIRR 9	Spruce sawdust not classified as a coal.
CIRR 15	Line Creek bituminous (no finer ranking done).

Table 8-1 Moisture analysis method used in responding to round robins

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																									
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
1					*			*							*	+	+	*	*	*	*		
2		*			*							*	*		*		+	+		+			
3			*	*				*							+	+	+	+	+				
4									*						+	+	+	+					
5					*	*													*	*	*		
6									*		*				+	+	+		+	*			
7			*		*	*	*	*			*		*	*	*	+	*	*			*		
8												*	*				+	+	+		*		
9	*	*	*												+			*		*			
10									*	*						+	+	+		*	+		
11															+	+	*	*	+		+		
12	*	*	*	*	*	*	*	*	*	*	*				1	1	1	1	1	1	1		
13			*	*			*				*				*	+		*	*	+			
14	*	*	*	*	*										+	+	+	+	+	*			
15			*	*	*	*	*	*	*	*	*	*			+	+	+	+	+	+	+		
16	*		*	*	*		*			*					+	+		+			+		
17				*	*						*				+	+		*		*			
18															+	+	+	+	+	+	+		
19									*											*			
20	*	*							*			*			*	+	+	+		+			
21										*	*	*			+	*	*	*	+				
22															*	+	+	+	+	+			
23					*		*	*	*				*		+		+	*			*		
24															+	+	+						
25										*	*				+	+	+	+	+				

Table 8-1 (cont'd)

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																								
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
26			*	*	*								*		+	*		+	+	*	*		
27								*	*	*	*		*		+	+		*	*	+	+		
28															+	+	*		+	*	*		
29							*								+	*	+	+	+	+	+		
30				*	*			*						-	*	*	*	*	+		+		
31		*													+	+	+	+	+	+	*		
32	*	*		*	*		*								+	+	*	+	+		+		
33															+	+	+	*	+		+		
34	*														+	+	+	*	+	+	+		
35	*					*										+	+	+	+	+	+	+	
36	*			*		*	*		*		*	*			*	+	+	+	+	*	+		
37	*														+	+	+	+	*	+	+		
38	*	*		*		*	*	*				*	*	*	+	+	+	*	+	+	+		
39	*	*	*	*	*	*	*								+	+		*					
40	*	*	*	*	*	*	*						*	*	*	*	*	*	+	+	+		
41	*	*	*	*	*	*	*				*							*		*			
42	*	*	*	*	*	*	*								+	+	+			+			
43	*	*	*	*	*	*	*					*			+	+	+	+	+	+	*		
44	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
45	*	*	*	*	*	*	*							*	+	+	+	+	+	+	+		
46	*	*	*	*	*	*	*								*	+	+	*	*	*	*		
47	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
48	*	*	*	*	*	*	*								+	+	+	+	+		+		
49	*	*	*	*	*	*	*								1	1	1	*	*	1	1		
50	*	*	*	*	*	*	*					*	*	-			-	*	*	*	+		
# of labs	27	30	29	26	25	31	28	40	39	42	38	39	39	42	38	43	40	32	43	34			

Table 8-2 Ash analysis method used in responding to round robins

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																									
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
1					*			*							*	+	+	*	*	*	*		
2		*			*							*	*		*		+	+		+			
3			*	*				*							+	+	+	+	+				
4									*						+	+	+	+					
5					*	*													*	*	*		
6									*		*				1	1	+		+	*			
7			*		*	*	*	*			*		*	*	*	+	*	*			*		
8												*	*				+	+	+		*		
9	*	*	*												+			*		*			
10									*	*						+	+	+		*	+		
11															+	+	*	*	+		+		
12	*	*	*	*	*	*	*	*	*	*	*				1	1	1	1	1	1	1		
13			*	*			*				*				*	+		*	*	+			
14	*	*	*	*	*										+	+	+	+	+	*			
15			*	*	*	*	*	*	*	*	*	*			+	+	+	+	+	+	+		
16	*		*	*	*		*			*					+	+		+			+		
17				*	*						*				+	+		*		*			
18															+	+	+	+	+	+	+		
19									*											*			
20	*	*							*			*			*	+	+	+		+			
21										*	*	*			+	*	*	*	+				
22															*	+	+	+	+	+			
23					*		*	*	*				*		+		+	*			*		
24															+	+	+						
25										*	*				+	+	+	+	+		+		

Table 8-2 (cont'd)

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																								
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
26			*	*	*								*		+	*		+	+	*	*		
27								*	*	*	*		*		+	+		*	*	+	+		
28															+	+	*		+	*	*		
29							*								+	*	+	+	+	+	+		
30				*	*			*							*	*	*	*	+		+		
31		*													+	+	+	+	+	+	*		
32	*	*		*	*		*								+	+	*	+	+		+		
33															+	+	+	*	+		+		
34	*														+	+	+	*	+	+	+		
35	*					*										+	+	+	+	+	+	+	
36	*			*		*	*		*		*	*			*	+	+	+	+	*	+		
37	*														+	+	+	+	*	+	+		
38	*	*		*		*	*	*				*	*	*	+	+	+	*	+	+	+		
39	*	*	*	*	*	*	*								+	+		*			+		
40	*	*	*	*	*	*	*						*	*	*	*	*	*	+	+	+		
41	*	*	*	*	*	*	*				*							*		*			
42	*	*	*	*	*	*	*								+	+	+			+			
43	*	*	*	*	*	*	*					*			+	+	+	+	+	+	*		
44	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
45	*	*	*	*	*	*	*							*	+	+	+	+	+	+	+		
46	*	*	*	*	*	*	*								*	+	+	*	*	*	*		
47	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
48	*	*	*	*	*	*	*								+	+	+	+	+		+		
49	*	*	*	*	*	*	*								1	1	1	*	*	1	1		
50	*	*	*	*	*	*	*					*	*					*	*	*	+		
# of labs	27	29	29	26	25	31	28	40	39	42	38	39	39	44	37	42	42	30	41	34			

Table 5

Table 8-3 Volatile matter analysis method used in responding to round robins

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																								
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
**********	**********				**********				**********				**********				**********						**
1					*			*							*	+	+	*	*	*	*		
2		*			*							*	*		*		+	+		+		
3			*	*				*							+	+	+	+	+			
4									*						+	+	+	+				
5					*	*													*	*	*	
**********	**********				**********				**********				**********				**********						**
6									*		*				1	+	+		+	*		
7			*		*	*	*	*		-	*	-	*	*	*	+	*	*			*	
8												*	*				+	+	+		*	
9	*	*	*												+			*		*		
10									*	*						+	+	+		*	+	
**********	**********				**********				**********				**********				**********						**
11															+	+	*	*	+		+	
12	*	*	*	*	*	*	*	*	*	*	*				1	1	1	1	1	1	1	
13			*	*			*				*				*	+		*	*	+		
14	*	*	*	*	*								-		+	+	+	+	+	*		
15			*	*	*	*	*	*	*	*	*	*			+	+	+	+	+	+	+	
**********	**********				**********				**********				**********				**********						**
16	*		*	*	*		*			*					+	+		+			+	
17				*	*						*				+	+		*		*	+	
18															+	+	+	+	+	+	+	
19									*											*		
20	*	*	-	-	-	-	-	-	*	-	-	*	-	-	*	-	-	-	-	-	-	
**********	**********				**********				**********				**********				**********						**
21										*	*	*			+	*	*	*	+			
22															*	+	+	+	+	+		
23					*		*	*	*				*		+		+	*			*	
24															+	+	+					
25										*	*				+	+	+	2	+		+	
**********	**********				**********				**********				**********				**********						**

Table 8-3 (cont'd)

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																								
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
26			*	*	*								*		+	*		+	+	*	*		
27								*	*	*	*		*		+	+		*	*	+	+		
28															+	+	*		+	*	*		
29							*								+	*	+	+	+	+	+		
30				*	*			*							*	*	*	*	+		+		
31		*													+	+	+	+	+	+	*		
32	*	*		*	*		*								+	+	*	+	+		+		
33															+	+	+	*	+		+		
34	*														+	+	+	*	+	+	+		
35	*					*										+	+	+	+	+	+	+	
36	*			*		*	*		*		*	*			*	+	+	+	+	*	+		
37	*														+	+	+	+	*	+	+		
38	*	*		*		*	*	*				*	*	*	+	+	+	*	+	+	+		
39	*	*	*	*	*	*	*							-	+	+		*			+		
40	*	*	*	*	*	*	*	-	-	-	-	-	*	*	*	*	*	*	+	+	+		
41	*	*	*	*	*	*	*				*							*		*			
42	*	*	*	*	*	*	*	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
43	*	*	*	*	*	*	*					*			+	+	+	+	+	+	*		
44	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
45	*	*	*	*	*	*	*							*	+	+	+	+	+	+	+		
46	*	*	*	*	*	*	*								*	+	-	*	*	*	*		
47	*	*	*	*	*	*	*								+	+	2	+	+	+	+		
48	*	*	*	*	*	*	*								+	+	+	+	+		+		
49	*	*	*	*	*	*	*								1	1	1	*	*	1	1		
50	*	*	*	*	*	*	*					*	*	-			-	*	*	*	+		
# of labs	27	30	28	25	23	30	27	37	37	38	35	36	36	40	36	41	36	27	39	32			

Table 8-4 Sulphur analysis method used in responding to round robins

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																									
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
1					*			*							*	2	2	*	*	*	*		
2		*			*							*	*	-	*		2	2		2			
3			*	*				*							+	+	+	+	+				
4									*						2	2	2	2					
5					*	*														*	*	*	
6									*		*				+	+	+		+	*			
7			*		*	*	*	*			*		*	*	*	+	*	*			*		
8												*	*				+	+	+		*		
9	*	*	*												+			*		*			
10									*	*						+	4	+		*	+		
11															-	2	*	*	2	2	2		
12	*	*	*	*	*	*	*	*	*	*	*				2	2	2	2	2	2	2		
13			*	*			*				*				*	2		*	*	2			
14	*	*	*	*	*	-									2	+	2	2	2	*	2		
15	-	-	*	*	*	*	*	*	*	*	*	*			+	+	+	+	+	+	+		
16	*		*	*	*		*			*					+	+		+					
17				*	*	-		-			*				+	+		*		*			
18															2	2	2	2	2	2	2		
19									*											*			
20	*	*							*			*			*	+	+	+		+			
21										*	*	*			+	*	*	*	+	-	-		
22		3	3			3									*	3	3	+	+	3			
23					*		*	*	*				*		2		2	*			*		
24															2	2	2						
25					-	-	-	-	-	*	*	-			2	2	2	+	2		2		

Table 8-4 (cont'd)

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																					
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
26			*	*	*								*		+	*		+	+	*	*		
27								*	*	*	*		*		+	+		*	*	+	+		
28															+	+	*		+	*	*		
29							*								+	*	+	+	+	+	+		
30				*	*			*							*	*	*	*	2		2		
31		*													+	+	+	+	+	+	*		
32	*	*		*	*		*								+	+	*	+	+				
33															2	+	+	*	+				
34	*														+	+	+	*	+	+	+		
35	*					*										+	+	+	+	+	+		
36	*			*		*	*		*		*	*			*	2	2	2	2	*	2		
37	*														+	+	+	+	*	+	+		
38	*	*		*		*	*	*				*	*	*	+	+	+	*	+	+	+		
39	*	*	*	*	*	*	*								+	+		*			+		
40	*	*	*	*	*	*	*	-	-	-			*	*	*	*	*	*	+	+	+		
41	*	*	*	*	*	*	*				*							*		*			
42	*	*	*	*	*	*	*								+	+	2			+			
43	*	*	*	*	*	*	*		-	-	-	*	-	-	-	-	-	-	-	-	*		
44	*	*	*	*	*	*	*								2	2	2	2	2	2	2		
45	*	*	*	*	*	*	*							*	+	+	+	+	+	+	+		
46	*	*	*	*	*	*	*								*	+	+	*	*	*	*		
47	*	*	*	*	*	*	*								2	2	+	2	2	+	+		
48	*	*	*	*	*	*	*								+	+	+	+	+		+		
49	*	*	*	*	*	*	*								1	1	1	*	*	1	1		
50	*	*	*	*	*	*	*	-	-	-	-	*	*	-	-	-	-	*	*	*	-		
# of labs	16	19	18	17	18	19	18	26	25	26	24	27	26	28	26	28	25	21	29	22			

note; 2=astm d3177 3=astm d1757 4=turbidimetry

Table 8-5 Calorific analysis method used in responding to round robins

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																							
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
1					*	-	-	*							*	+	+	*	*	*	*		
2		*			*							*	*		*		+	+		+			
3			*	*				*							+	+	+	+	+				
4									*						+	+	+	+					
5					*	*													*	*	*		
6									*		*				+	+	+		+	*			
7			*		*	*	*	*			*		*	*	*	+	*	*			*		
8												*	*				+	+	+		*		
9	*	*	*												+			*		*			
10					-				*	*						+	+	+		*	+		
11															+	+	*	*	+	-	+		
12	*	*	*	*	*	*	*	*	*	*	*				1	1	1	1	1	1	1		
13			*	*			*				*				*	+		*	*	+			
14	*	*	*	*	*										+	+	+	+	+	*			
15	-	-	*	*	*	*	*	*	*	*	*	*			+	+	+	+	+	+	+		
16	*		*	*	*		*			*					+	+		+					
17				*	*						*				+	+		*		*			
18															+	+	+	+	+	+	+		
19									*											*			
20	*	*							*			*			*	+	+	+		+			
21								-	-	*	*	*			+	*	*	*	+	-	-		
22															*	+	+	+	+	+			
23					*		*	*	*				*		+		+	*			*		
24											-				+	+	+						
25										*	*				+	+	+	+	+		+		

Table 8-5

CANSPECS Laboratory Number	CIRR/CANSPECS Sample Number																								
	2	3	4	5	6	7	8	10	11	12	13	14	16	17	18	19	20	21	22	23	24	25	26
26			*	*	*								*		-	*		+	+	*	*		
27								*	*	*	*		*		+	+		*	*	+	+		
28															+	+	*		+	*	*		
29							*								+	*	+	+	+	+	+		
30				*	*			*							*	*	*	*	+		+		
31		*													+	+	+	+	+	+	*		
32	*	*		*	*		*								+	+	*	+	+		+		
33															+	+	+	*	+		+		
34	*														+	+	+	*	+	+	+		
35	*					*										+	+	+	+	+	+	+	
36	*			*		*	*		*		*	*			*	+	+	+	+	*	+		
37	*														+	+	+	+	*	+	+		
38	*	*		*		*	*	*				*	*	*	+	+	+	*	+	+			
39	*	*	*	*	*	*	*								+	+		*			+		
40	*	*	*	*	*	*	*						*	*	*	*	*	*	+	+	+		
41	*	*	*	*	*	*	*				*							*		*			
42	*	*	*	*	*	*	*								+	+	+			+			
43	*	*	*	*	*	*	*					*			+	+	+	+	+	+	*		
44	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
45	*	*	*	*	*	*	*							*	+	+	+	+	+	+	+		
46	*	*	*	*	*	*	*								*	+	+	*	*	*	*		
47	*	*	*	*	*	*	*								+	+	+	+	+	+	+		
48	*	*	*	*	*	*	*								+	+	+	+	+		+		
49	*	*	*	*	*	*	*								1	1	1	*	*	1	1		
50	*	*	*	*	*	*	*					*	*	-	-	-	-	*	*	*	-		
# of labs	26	29	29	26	24	30	27	39	38	42	37	38	39	43	36	42	40	30	41	32			